COTTONSEED AND ITS PRODUCTS
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Vanaspati Research Advisory Committee
COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH
NEW DELHI
1954

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COTTONSEED AND ITS PRODUCTS

By

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FOREWORD

India is one of the major cotton growing countries in the world and her ginning industry yields as by-product about one million tons of cottonseed which can be utilized with benefit for the production of edible oil and other useful products.

Cottonseed has been used in India mostly as cattle feed. It is estimated that about 1,00,000 tons of cottonseed oil can be produced in India. Actual production, however, is only 5 per cent of this quantity and the target envisaged by the Planning Commission is 12,500 tons, representing about 12.5 per cent of the total, at the end of the first five-year period. We have hardly any industry for utilizing the hulls. Cottonseed has been commercially exploited in other countries and a large variety of products, besides the edible oil—paper, plastics, rayon, lacquers and explosives from linters; activated carbon and furfural from hulls; and cattle feed from seed cake—are obtained on an industrial scale.

Scientific and technological information on the utilization of cottonseed is extensive but scattered and the compiler of this pamphlet has rendered valuable service by collecting the information and presenting it in a concise manner with adequate references to the original sources. It is hoped that the publication would stimulate interest in the commercial utilization of a raw material which is itself a by-product of the cotton ginning industry.

S. S. BHATNAGAR

PREFACE

The present publication reviews the progress made in the utilization of cottonseed during the past 50 years and gives much valuable information on the use of cottonseed and its products in food technology. Mr. M. N. Krishnamurthi deserves praise for his enthusiasm and painstaking efforts in collecting and compiling from a number of sources the material for this publication. It is hoped that the publication would be of value both to scientific workers and to the industry.

Acknowledgement is due to Dr. G. T. Kale for his keen interest and valuable help in the preparation of the matter; to Dr. M. Swaminathan, Dr. M. Narayana Rao, Mr. M. V. Lakshminarayana Rao, Major N. V. R. Iyengar and Mr. A. N. Sankaran for reading through the manuscript and making a number of valuable suggestions; and to the Governing Board of the American Oil Chemists' Society for kind permission to publish some of the standard methods of analysis.

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CHAPTER I

THE ROLE OF COTTONSEED IN INDIAN ECONOMY

The cotton plant is cultivated primarily for the cotton fibre required for spinning yarn rather than for its seed. One of the first recorded references to cotton occurs in ancient Hindu *Puranas* about 1,500 years before the Christian era. Other sources indicate that, in the third century B.C., the army of Alexander the Great found cotton widely used by the Indian people and introduced this plant in the eastern and southern borders of the Mediterranean Sea, from where it later spread to Europe. Archaeological evidence obtained at Mohenjo-daro indicates that cotton was used in India as early as 3,000 B.C.

STATISTICAL REVIEW

Production and Trade

Reliable data on cottonseed production are not available. Hence cottonseed production is calculated from the lint production; even with regard to cotton lint, estimates obtained from different statistical organisations are found to vary rather widely. Trade estimates are generally higher than official ones. For estimating the cottonseed production, the U.S. Bureau of Census have adopted the ratio of seed to lint as 63:37. The figures obtained by using this ratio for India and other countries should be regarded as only approximate, since the ratio of seed to lint varies with the soil, climatic conditions and variety of the plant.

Figures of cottonseed production computed by using the above ratio from the estimated production of cotton lint in different countries in 1951-52 by the International Cotton Advisory Committee[4] are shown in Table 1.

Among the cotton growing countries, U.S.A. continues to be the largest producer in the world, with U.S.S.R. ranking second. Other major producers of cotton are India, China, Brazil, Egypt, Pakistan and Mexico. In recent years, the production in Brazil has fallen while that in Mexico has improved. As for future development, though there is unlimited scope for growing the finest varieties of cotton, the immediate world need is to enhance the yield per acre

TABLE 1—WORLD COTTONSEED PRODUCTION IN 1951-52

	Million metric tons
U.S.A.	6.34
Mexico	0.57
Canada	0.05
India	1.32
Iran	0.05
Pakistan	0.54
Syria	0.09
Turkey	0.26
Other Asian countries	0.13
Sudan	0.12
Belgian Congo	0.09
Egypt	0.70
French Africa	0.06
Mozambique	0.05
Nigeria	0.05
Uganda	0.13
Other African countries	0.06
Argentina	0.25
Brazil	0.84
Peru	0.17
Other South American countries	0.06
U.S.S.R.	1.59
China	1.25
Eastern Europe	0.04
Others	0.08
World Total	14.90

in areas where cultivation has already been established[3,5]. For example, India stands third among the important cotton growing countries and produces only 9 per cent of the world's output of cotton, on an acreage which is about 20 per cent of the world's acreage under this crop. According to the information obtained from the Indian Central Cotton Committee the yield per acre of cotton in India ranges from 60 to 90 lb. in the rainfed areas, and from 180 to 200 lb. per acre in irrigated areas; in U.S.A. and in Egypt it is 267 lb. and 300 lb. per acre respectively[6].

From the position of world supply, Egypt was the largest pre-war source of cottonseed. Next in the order of importance were Uganda

and Anglo-Egyptian Sudan. Other prominent exporters were China, Brazil and Peru. During the war and post-war periods, exports declined considerably, the cause for the decline being : fall in production of exporting countries, need for conserving supplies of edible oil for domestic consumption, currency difficulties, and shipping limitations. In recent years, the bulk of exports came mainly from Uganda and Sundan[3,5].

Before the war the foremost cottonseed buyer was the United Kingdom, which absorbed 80 per cent of the world's exports. Japan and Chile followed next, but their intake was relatively small. In the later war years, the bulk of supplies went to Egypt which was formerly the leading exporter. In 1950 the United Kingdom was again in the leading position, the quantity being a little less than one-fourth of the average imported into U.K. in 1934-38[3,5].

Table 2 shows world and Indian totals of acreage under cotton and production of cottonseed for 1938-39 and 1947-52[3,7-9].

India grows a large number of varieties of cotton. Table 3 shows the acreage and production of the main varieties[8].

Among the Indian varieties of cotton, *Oomras* is the most important. With the increasing demand for long staple cotton from Indian textile mills, the trend has been to grow the more advantageous and better yielding American variety wherever possible. This variety constitutes about 10 per cent of the total production. But due to a higher linter content as compared with that of *deshi*, it is relatively unpopular as a source of cottonseed and therefore fetches a lower price. However, the American variety has 20-22 per cent oil and 10-14 per cent linters and is preferred by oil millers for seed crushing in comparison with the *deshi* seed which has an average of 17-19 per cent oil and 2.5 to 5 per cent linters[10].

TABLE 2—ACREAGE UNDER COTTON AND PRODUCTION OF COTTONSEED

	Area (million acres)		Production of cottonseed (million metric tons)	
	World total	Indian total	World total	Indian total
1938-39	75.4	23.5	12.19	2.10
1947-48	58.0	10.7	10.01	0.87
1948-49	60.9	11.1	11.32	0.76
1949-50	66.9	12.17	11.94	0.90
1950-51	n.a.	14.6	11.10p	1.02
1951-52	n.a.	16.2	13.92	1.07

n.a. = not available. p = preliminary estimates.
Indian figures for 1938-39 include those for Pakistan.

Details of area under cotton and production of cottonseed in different States in the Indian Union[8] for the year 1951-52 are shown in Table 4.

The first record of the crushing of cottonseed dates back to the early times. In ancient Hindu scripts it is reported that cottonseed oil was used in medicine and cottonseed meal as cattle-feed. U.S.A.

TABLE 3—ACREAGE UNDER COTTON VARIETIES AND PRODUCTION OF COTTONSEED IN INDIA DURING 1951-52 (BY VARIETIES)

Variety	Area (thousand acres)	Production of cottonseed (thousand metric tons)
Bengals	1,156	142.70
American	910	95.69
Oomras	4,503	333.74
Broch	902	42.88
Surti	407	19.21
Dholleras	1,780	71.69
Others	6,555	369.01
	16,213	1,074.90

TABLE 4—AREA UNDER COTTON AND PRODUCTION OF COTTONSEED IN 1951-52

States	Area (thousand acres)	Production (thousand metric tons)
Bombay	3,981	191.5
Madhya Pradesh	3,295	267.5
Hyderabad	3,107	155.7
Madras	1,796	139.6
Madhya Bharat	1,415	63.8
Saurashtra	935	35.0
Punjab	512	83.7
Rajasthan	341	26.0
PEPSU	301	51.0
Uttar Pradesh	193	21.3
Mysore	134	24.0
Other States	203	15.8
	16,213	1,074.9

is now the foremost country in which cottonseed crushing is carried out on an extensive scale. The first commercial mill was started in U.S.A. in 1834. It was rather crude and inefficient. However, the development of machinery for the removal of linters and hulls in 1870 contributed largely to the growth of large scale cottonseed oil industry. In 1951-52 the production of cottonseed oil in U.S.A reached a peak level of 1,950 million lb., an increase of 60 per cent over 1950-51 level. This figure[5] exceeds the previous record of 1,900 million lb. in 1937-38.

U.S.S.R. has made strenuous efforts during recent years to utilize domestic resources to the fullest extent. In China, cottonseed crushing is carried on to a large extent by primitive methods. In the past few years, Brazil, Egypt and Pakistan have expanded cottonseed milling markedly. In India, although there are over 1,100 big oil mills, the estimate of the total quantities of cottonseed crushed and the number of mills crushing this raw material for the past few years are not exactly known. The Planning Commission has estimated the production of cottonseed oil at 5,000 tons during 1949-50 from about 50,000 tons of cottonseed out of a total of about one million tons. The target fixed in the first Five Year Plan is 12,500 tons of cottonseed oil in 1955-56.

In order to encourage the growth of cottonseed oil industry in India, the Planning Commission has recommended the following measures[11] :

(i) Duty-free import of delinting machinery;
(ii) licences for the expansion of oil mills or the establishment of new mills to units crushing cottonseed;
(iii) provision of special facilities for obtaining activated carbon;
(iv) levy of differential excise duty on vanaspati produced entirely from cottonseed oil;
(v) education of the farmer in favour of using cottonseed cake as cattle-feed ;
(vi) reduction in railway freight on cottonseed; and
(vii) reduction of export duty on cottonseed oil and linters.

A significant post-war development in world trade in oils and oilseeds has been the movement of oils rather than of oilseeds, mainly due to the expansion of oil milling capacity in oilseed producing countries. Among the principal exporting countries in cottonseed oil, Brazil maintains its pre-war status, but on a much reduced scale. Before World War II, U.S.A. was a net importer of oil, but during the war the import and export trade widely fluctuated and in 1947-49

the export showed an upward trend. With the rise in exports from U.S.A., countries such as U.K. and Canada commenced receiving increased supplies of cottonseed oil by 1949, partly under the European Recovery Programme. The other exporters of cottonseed oil were Argentina, Egypt and China, though their exports fluctuated widely [3].

INDUSTRIAL USES

Linter

In addition to the true fibre or lint, the cottonseed bears a large number of short fibres, called linters or fuzz. These linters form an average of $2\frac{1}{2}$ per cent in *deshi* seed and 10 per cent in the American variety. Linters form an important by-product of the cottonseed oil industry in U.S.A. The U.S. Department of Agriculture classifies the linters into seven grades according to fibre length, colour, character and amount of trash. The highest grade of linters obtained from the 'first cut' is spun for making surgical cotton and in such products as twines, wicks, carpets, etc. The intermediate grades (mill run) are used for stuffing mattresses, pillows, cushions and for felts. The last grade or the 'second cut' is used in chemical industries for the manufacture of smokeless powder, rayon, paper, plastics, cellulose lacquer, etc.

In U.S.A. the value of linters produced exceeds 52 million dollars annually. In India, if all the cottonseed produced is scientifically processed and utilized, the value of linters will be a substantial addition to the profits of the industry. It is estimated that the present output of one million tons of cottonseed will yield about 25,000 tons of linters.

Hull

For many years, the cottonseed hulls were used as fuel. Hulls require large storage space, since the material occupies 30 times as much space as coal of equivalent fuel value. Moreover, hull storage involves fire hazard. It was in 1885 that cottonseed hulls began to be used fairly extensively as a roughage for live-stock, and in subsequent years a preferred market was created in U.S.A. for cake of lower protein content having a large amount of hulls in it.

There are many other uses for hulls which find important applications in chemical and other industries, such as the manufacture of activated carbon, plastics, xylose and furfural. Markley[12] found that cottonseed hull bran yields 25.33 per cent furfural. Destructive distillation of cottonseed hulls produces a viscous brown

tar readily miscible with kerosene. Hopkins[13] prepared an efficient mosquito larvicide from a mixture of 3 volumes of cottonseed hull tar and one volume of kerosene.

Cottonseed Flour

The cottonseed flour can be used for clarifying sugar solutions (*U.S. Pat.*, 2,501,272). It is also used in the manufacture of adhesives, cattle-feed and bakery products.

Cottonseed Pigments

The yellow pigment in cottonseed may be used as a dye for silk and wool, but no commercial process appears to have been developed as yet. Since gossypol is toxic when used in food products, it can be used as an antioxidant in non-edible products, such as plastics, rubber and drying oils. Gossypol with mustard and in the presence of copper or manganese, stimulates seed germination.

Cottonseed Oil Foots

Recent researches have shown that Indian cottonseed oil foots contain valuable fatty acids. In a new method, Bhushan *et al.*[14] hydrolysed cottonseed oil foots at 100-110 lb./sq. in. for 6-12 hr. in an autoclave. After cooling, the emulsion was boiled with excess dil. sulphuric acid and the fatty acid layer separated and dried at 110°C. after washing free of mineral acid. The resultant dark viscous mass was twice distilled with superheated steam (240-260°C.) at 400-420 mm. pressure. The yield of total fatty acids was 46.9 per cent. The fatty acids consist of 50.9 per cent saturated (mainly palmitic acid) and 49.1 per cent unsaturated acids, which on hydrogenation gave technically pure stearic acid. The residual dark green brittle shining stearic pitch left in the distillation still is used for caulking ship decks, as insulating material, for water-proofing and for making varnishes and anti-rust paints. The softer stearic pitch is used as a lubricant for heavy steal plate rollers.

Herbert (*U.S. Pat.*, 2,435,456) patented a process for the production of fatty acids from cottonseed oil foots. The process consisted in treating with sodium hydroxide, 0.05-0.5 per cent potassium persulphate or sodium hypochlorite during the saponification and distilling the separated soap stock under reduced pressure after a prior acid treatment. Tu and his co-workers[18] investigated cottonseed oil foots to obtain motor fuel and allied products. In India, Bhushan *et al.*[14] studied the pyrolysis of Indian cottonseed oil foots. Under optimum conditions the foots yield 21 per cent crude oil (cal. val., 17,400 B.t.u.). When the oil is distilled, gasoline (31-35 per cent)

and kerosene (43-50 per cent) were obtained. The gasoline has a calorific value of over 19,000 B.t.u. while kerosene (b.p., 225°-325°) had a value of 19,200 B.t.u.

BIBLIOGRAPHY

1. *Cottonseed and its Products* (National Cottonseed Products Assoc. Inc. Memphis, Tenn.), 1950.
2. Bailey, A.E., *Cottonseed and Cottonseed Products* (Interscience Publishers, Inc., New York), 1948.
3. *Vegetable Oils and Oilseeds* (Commonwealth Economic Committee), 1952, 18-29
4. *Commerce,* **86** (1953), 290
5. *Paint Oil Col. J.,* **121** (1952), 163
6. Address by P. R. Deshmukh at sixty-seventh meeting of the Indian Central Cotton Committee, 1953
7. *Commerce,* **85** (1952), 770, A51-52
8. *Agric. Situat. India,* Directorate of Economics & Statistics, Ministry of Food & Agriculture, Govt. of India, **7** (1952), 518-19
9. *Agric. Situat. India,* Directorate of Economics & Statistics, Ministry of Food & Agriculture, Govt. of India, **7** (1952), 226
10. Turner, S. H., *Report on the Edible Vegetable Oil Resources of Pakistan,* 1948
11. *Programmes of Industrial Development,* Planning Commission of the Government of India (1951-56)
12. Markley, K.S., *J. Amer. Soc. Agron.,* **20** (1928), 1102-07
13. Hopkins, G. H. E., *E. Afr. med. J.,* **20** (1943), 41-45
14. Bharat Bhushan, Phillips, J. P. & Gulati, K. C., *J. sci. industr. Res.,* **12B** (1953), 38-40
15. Nanjundiah, C., *Indian Cott. Gr. Rev.,* **6** (1952), 3
16. *Industr. Engng Chem. (News),* **18** (1940), 193
17. Leahy, *J.F., Sth. Pwr Ind.* **59** (1941), 44
18. Tu, C. M. & Pan, F. Y., *J. Chem. Engng , China,* **3** (1936), 231

CHAPTER II

COMPOSITION OF COTTONSEED

Commercial cottonseed as obtained from the gin is composed of the following physical parts: (a) the linters which are the short fibres attached to the hull and not removed in the ginning process, (b) the hull which is a dark coloured shell, and (c) the kernel which is the inner part of the seed. Tables 5 and 6 show the ultimate and proximate analysis of whole Indian Cottonseed[1,2].

TABLE 5—ULTIMATE COMPOSITION OF WHOLE INDIAN COTTONSEED

	Linters	Delinted whole seed		Oil
		hull %	kernel %	%
Indigenous variety	4.0	44.0	56.0	19.2
American variety	13.8	44.0	56.0	22.0

TABLE 6—PROXIMATE COMPOSITION OF SOME IMPORTANT VARIETIES OF COTTONSEED

	Moisture %	Ash %	Crude protein %	Albuminoid %	Fat %	Fibre %	Carbo-hydrate %
Cambodia	8.61	4.28	19.19	18.65	17.11	23.61	27.33
Northerns	8.52	3.66	19.12	17.70	19.81	22.14	26.75
Westerns	8.60	3.84	19.78	18.49	17.49	16.75	33.54
Tinnevelies	8.76	3.41	17.81	16.25	17.40	22.84	29.78
Uppam	8.43	3.62	16.29	14.79	16.96	24.37	30.32

The relation between variety, growth and environment to the oil, carbohydrate, fibre, ash and protein contents of the seed has been investigated by several workers. Some reported that a few varieties contain meats with a somewhat higher oil content than the others. Grindley[3] observed in his study that the oil content increases rapidly from the thirty-fifth day of maturity after flowering until the sixtieth day and then remains more or less constant. The most rapid increase in oil content was between the forty-first and the fifty-first days. During this period, the protein as well as ash also increased steadily in proportion to the weight of the seed, while the crude fibre showed a sharp increase shortly before maturity after flowering. Grindley[3]

further noticed that the oil at first formed was highly acidic and contained high proportion of unsaponifiable matter.

The substance[4] commonly found in cottonseed kernel are: Cellulose; pectin; starch; pentosans and pentoses; sucrose; raffinose and such other sugars; glucosides, saponins, etc.; protein and other nitrogen compounds; lipids, fats and oils; phosphatides and phosphorus compounds; sterols (phytosterol, silosterol, etc.); waxes and higher alcohols; pigments (gossypol), etc.; organic acids (citric and malic acids); vitamins and antioxidants (tocopherols, etc.); enzymes and hydrocarbons.

Cottonseed Pigments

The high pigment content of cottonseed kernels gives rise to many technical problems not encountered in processing of other oilseeds. The principal pigment, gossypol ($C_{30}H_{30}O_8$), which is light yellow in colour, is a phenolic compound occurring in a concentration of 0.4-2 per cent on the weight of the kernel[4,5]. Appreciable amounts of active gossypol, if present in the meal, give toxic physiological effects[4]. The intensity of the colour of the expressed oil varies according to the method of expression. Normally, an average of 0.05 per cent of gossypol is found in crude oil. In matured seeds, there is a direct relationship between the oil and gossypol content. The ratio of oil to gossypol ranges from 35-55 parts oil per one part of gossypol[4]. Smirnova[6] adduced evidence to show that genetic factors exert a significant influence on the gossypol content of cottonseed independent of the environmental factors.

Later investigations on cottonseed pigments have resulted in the isolation of three other pigments in addition to gossypol. They are: (a) gossypurpurin—a dark purple pigment, (b) gossycaeurlin—a blue coloured pigment found only in cooked cottonseed meats, and (c) gossyfulvin — an orange yellow pigment. The gossypol in the glands constitutes about 35-50 per cent and gossypurpurin about 1 to 3 per cent on the weight of glands[7]. Still more recently evidence has been obtained of the presence of 11 other pigments in addition to gossypol in pigment glands[8].

It has been observed that when cottonseed is stored at room temperature there is an increase in "red gossypol" and free fatty acid content [9,10]. These changes may be prevented by storing the seed at moisture content of 8 per cent or less at a temperature of 1°C. or below in a closed chamber[10,23]. At storage temperatures of 38°, 77° and 85°F. the gossypurpurin increases in proportion to the temperature and the length of time, while the gossypol content decreases[24].

Carbohydrates

The more important components, next to oil and protein, in cottonseed kernel are carbohydrates. Grindley[3] reported that the carbohydrates in cottonseed are at a maximum level between the twenty-first to the thirty-fourth day and between the forty-first and the fifty-first day of maturity after flowering, the carbohydrate content shows a decrease. It is therefore likely that at this falling period, the carbohydrate is being converted into fatty material. The principal component of the carbohydrate fraction of the cottonseed kernel is raffinose ($C_{18}H_{32}O_{16}$). According to Hudson and Harding[4] an average of 6-8 per cent of raffinose is present in the meal and it is the cheapest and the most convenient source of raffinose. In commercial preparations an yield of 2-4 per cent of raffinose from the meal is obtained.

Fatty Acids

Cottonseed oil when used for edible purpose supplies about 270 cal./oz. The essential constituent which influences the growth and functioning of the body are the unsaturated fatty acids which occur in a high percentage in cottonseed oil. Indian samples of cottonseed oil according to Hilditch[11] contain: oleic acid, 22.9-29.6; linoleic acid, 45.3-50.4; palmitic acid, 19.6-23.4; stearic acid, 1.1-2.7; myristic acid, 1.4-3.3; and arachidic acid, 0.6-1.3 per cent.

Table 7 gives the important physical and chemical characteristics of cottonseed oil according to the specification of the American Oil Chemists' Society[12].

TABLE 7—PHYSICAL AND CHEMICAL CHARACTERISTICS OF COTTONSEED OIL

Sp. gr.$^{25°}$	0.916-0.918
$n^{25°}$	1.463-1.472
Iodine value	99-113
Thiocyanogen value	61-69
Sap. value	189-198
Unsaponifiable matter, %	below 1.5
Titer (°C.)	30-37
Acetyl value	7.5-12.5
Reichert-Meissl value	below 1
Polenske value, %	„
Saturated acids, %	22
Unsaturated acids, %	76
Special characteristics	Positive Halphen test

Stansbury[13] and his co-workers have shown that cottonseed oils obtained from seeds of different varieties grown under different environmental conditions vary widely in iodine value and that the percentages of linoleic, oleic and saturated acids are closely correlated with the iodine value.

Phosphorus Compounds

Cottonseed has a higher content of total phosphorus, phosphatides and phytin-phosphorus than any other oilseed[4]. The phosphatides are found in both oil and meal, the relative amounts depending on the methods of extraction. According to Goldovskii and Lishkevich[4] 10 per cent of the phosphatides occur in the cottonseed oil and the remainder in the cake. Among the common phosphatides, the lecithin fraction constitutes one-third of the total, the rest being cephalin.

Sterols

The unsaponifiable portion of the oil consists of sterols, tocopherols, hydrocarbons, etc. According to Kaufmann[14] crude oil contains up to 1.6 per cent sterols which decreased during refining. A crude sterol fraction from cottonseed oil contains 5 per cent of pro-vitamin D.

Antioxidant

In the extraction of oil from cottonseed, oil soluble vitamins A, D, E and K, and gossypol accompany the oil, leaving water soluble vitamins, like the vitamin B complex and vitamin C, in the residual meal. The vitamin B complex content of cottonseed flour is given in Chapter V.

Gossypol is one of the most active of all the naturally occurring antioxidants of vegetable origin but its use is restricted in edible products due to its toxic properties. The other natural antioxidant present in cottonseed oil is tocopherol. Its effect is enhanced by the presence of phosphatides which act as synergists. Fisher[15] found that crude cottonseed oil cohtained 0.076 per cent α- and 0.034 per cent γ-tocopherol, while refined cottonseed oil contained 0.065 per cent α- and 0.026 per cent γ-tocopherols; α-tocopherol is identical with vitamin E. More recently, Weisler *et al.*[16] reported about 1.0 per cent of the total tocopherols in cottonseed oil to be δ-tocopherols. Mack *et al.*[17] investigated the relationship between the tocopherol content, fatty acid composition and stability of cottonseed oil. They noticed an increase in the tocopherol content with an increase in the unsaturation of glycerides. Besides these two antioxidants, Golumbic[18] found the presence of other antioxidants in cottonseed oil. They are

similar to chroman-5, 6-quinones in their chemical behaviour. Crude cottonseed oil which is considerably more resistant to oxidation than refined oil probably owes its stability to the presence of all these naturally occurring antioxidants.

Dollear[19] reported that the stability of refined cottonseed oil can be increased by hydrogenation or by the addition of antioxidants. Of the various antioxidants which improve the keeping quality, propyl gallate was found to be the best. Nordihydroguairetic acid norconidendren[20] also gave satisfactory results. John *et al.*[21] recommended the storage of oil in amber coloured, vacuum tight glass bottle to improve its keeping quality.

Proteins

The composition of the protein is discussed under Chapter V.

Lipase

Olcott and Fontaine[22] reported that the lipolytic activity in cottonseed develops during germination. The lipase was most effective at *p*H 6-9 and is completely absent in dormant seeds.

BIBLIOGRAPHY

1. Harwalker, V. R., Soni (Miss), P. & Achaya, K. T., *Indian Soap J.*, **18** (1952), 202
2. Official Communication of the Government Agricultural Chemist, Agriculture College and Research Institute, Coimbatore, India
3. Grindley, D. N., *J. Sci. Fd Agric.*, **1** (1950), 147
4. Guthrie, J. D., Hoffpauir, C. L., Stansbury, M. F. & Reeves, W. A., *Survey of Chemical Composition of Cotton fibres, Cottonseed, Peanuts and Sweet potatoes—A Literature Review* (A.I.C. 61), Rev. March 1949. Issued by the United States Department of Agriculture, Agriculture Research Administration—Bureau of Agricultural and Industrial Chemistry
5. Hall, C. M., O'Connor, R. T., Castillon, L. E. & Curet, M. C., *J. Amer. Oil Chem. Soc.*, **24** (1947), 276-283
6. Bailey, A. E., *Cottonseed and Conttonseed Products* (Interscience Publishers, Inc., New York), 1948
7. Castillon, L. E., Hall, C. M. & Boatner, C. H., *J. Amer. Oil Chem. Soc.*, **25** (1948), 233-236
8. Markley, K. S., *Oil Mill Gaz.*, **51** (1947), 27-31
9. Boatner, C. H., O'Connor, R. T., Curet, M. C. & Samuels, C. S., *J. Amer. chem. Soc.*, **69** (1947), 1268-1273
10. Stansbury, M. F. & Guthrie, J. D., *J. agric. Res.*, **75** (1947), 49-61
11. Hilditch, T. P., *The Chemical Composition of Natural Fats* (Chapman & Hall, London) 2 Edn, 1947
12. Mehlenbacher, V. C., Editor, *Amer. Oil Chem. Soc.*, 1946, *Official and and Tentative Methods A.O.C.S. Table I.*, 1-46
13. Stansbury, M. F. & Hoffpauir, C. L., *J. Amer. Oil Chem. Soc.*, **29** (1952), 53-55
14. Kaufmann, H. P., *Fette u Seif.*, **48** (1941), 53-59
15. Fisher, G. S., *Industr. Engng Chem. (Anal.)*, **17** (1945), 224
16. Weisler, L., Robeson, C. D. & Baxter, J. D., *Industr. Engng Chem. (Anal.)*, **19** (1947), 906
17. Mack, C. H., Moore, R. N. & Bickford, W. G., *J. Amer. Oil Chem. Soc.*, **29** (1952), 14-15

18. Golumbic, Calvin, *J. Amer. chem. Soc.*, **64** (1942), 2337-40
19. Dollear, F. G., *Potato Chipper*, 1952—(A.I.C. 319, Supplement No. 2, 40, 12, 1952)
20. Annual Report of U. S. Dept. of Agric. Bureau of Agric. & Industr. Chem., 1951, 16-17
21. John, E. W., McConnell & Williams B. Essen, Jr., *J. Amer. Oil Chem. Soc.*, **24** (1947), 6-13
22. Olcott, H. S. & Fontaine, T. D., *J. Amer. chem. Soc.*, **63** (1941), 825
23. Pons, Jr. W. S., Murray, M. D., O'Connor, R. T. & Guthrie, J. D., *J. Amer. Oil Chem. Soc.*, **25** (1948), 308-313
24. Castillon, L. E., Hall, C. M., O'Connor, R. T. & Miller, C. B., *J. Amer. Oil Chem. Soc.*, **26** (1949), 655-9
25. *Cottonseed and its products* (National Cottonseed Products Association Inc. Memphis 3, Tenn), 4 Edn, 1950

CHAPTER III

COTTONSEED STORAGE

Considerable stocks of the seed have to be built up at the time of harvesting so that the raw material may be available to the oil milling industry all the year round. Seed deterioration during storage due to heating and insect attack constitutes a major problem to the processors. Heating takes place in seeds of high moisture content and is due to the enzymatic activity within the seeds. The heat generated cannot be readily dissipated due to the insulating effect of lint on the seed. As a result the quality and yield of processed products is affected. For example, cottonseed which has been damaged by heating during storage yields less oil than fresh cottonseed and the oil which is obtained contains an increased percentage of free fatty acids. In commercial storage, heating of the seeds is minimised by one of the following procedures: (1) pre-drying the seeds prior to storage, (2) forced aeration during storage, (3) stacking bagged seeds in such a manner that natural circulation of air in the interspaces is facilitated and finally, (4) chemical inhibition of biological activity within the seeds[2].

Two types of storage units are common in commercial practice; they are ware-houses and silos. In modern silo design efficient air circulating devices and automatic indicating and controlling instruments are provided so that the amount of air, moisture and temperature in the silos may be regulated and controlled as required.

A number of investigators have studied the aeration of seed under storage to reduce the heating of seeds by dissipating away the heat of respiration. Karon[3] and co-workers have observed that the relative humidity of air used either increases or decreases the moisture content of seed. Any increase in moisture content stimulates further respiration resulting in increased heating. According to Jensen *et al.*[4] prolonged aeration reduces moisture content, but if the seed contains high initial moisture or free fatty acids the production of heat is accelerated. The effect of flash heating on storability of seed was investigated by Whitten[5]. He found that flash heating and

drying keeps cottonseed in good condition for over six months if the initial condition of the seed is good. Lyman[6] and his colleagues found that the exposure of moist cottonseed to dielectric heating for a period of 2-5 minutes effectively reduces the moisture and destroys the enzymes responsible for the formation of free fatty acids.

Chemical Inhibition

Although the rise of temperature is controlled by the circulation of air or by mechanical cooling procedures, the seed may yet deteriorate if the lot exhibits a tendency to heat when stored in bulk. The use of chemical inhibitors presents an interesting approach to such problems of heating and consequent deterioration. Altschul *et al.*[7] studied the effect of inhibitors on the lipolysis and respiration of cottonseeds. They found that ammonia treatment inhibited both respiration and lipolysis of mature seeds; with immature seeds, however, lipolysis was stimulated. The vapours of Nacconol NR (alkyl-aryl-sodium sulphonate) and 2′-methyl-l-maleanil inhibited lipolysis only when respiration was stimulated. For stimulating respiration fungicides and germicides such as Emulsol 607 (alkaline emulsified oils) and butyl malemide may be employed. Deterioration due to biological activity in cottonseed containing about 15 per cent moisture is inhibited by treatment with 0.2—1 per cent of a mixture containing 10 parts of propylene glycol dipropionate (P.G.D.P.) and one part 1, 3-dimethyl 4- and 6-*bis*-chloromethyl benzene or -xylene (*U.S. Pat.*, 2,571,095). Jensen *et al.*[8] found that treatment of seed at the rate of 30 lb. of the mixture eliminated heating during a 73-day storage period, and with drying to low moisture content, the development of free fatty acids was inhibited for 52 days. They also observed that seeds at the beginning of the season were high in moisture content and those harvested late showed some break-down of protein. Condon and his associates[9] showed that ethylene chlorohydrin and propolene chlorohydrin inhibited free fatty acid formation and heating. A concentration of 0.19 per cent of the chemicals on the dry weight of the seed gave the best results. Cottonseed up to 30 per cent moisture content and containing natural enzymes responded to this chemical treatment (*U.S. Pat.*, 2,584,972). The method consists in spraying the seed with liquid ethylene chlorohydrin in a concentration of about 0.2-1.0 per cent on the dry weight of the seed.

Viable Seeds

Cottonseed having a moisture content of 8-11 per cent is often stored without loss of viability for periods as long as two years in most parts of U.S.A. Lambou *et al.*[10] studied the effect of chemical

treatment on cottonseed of high moisture content with regard to viability and growth. Solution of 4, 6-*bis*-chloromethyl xylene in propylene glycol dipropionate in the ratio of 1:8, stimulated both viability and growth. Further tests proved that the treatment did not merely accelerate germination but actually resulted in a higher percentage of germination. An analysis of the sample for moisture and free fatty acids after $6\frac{1}{2}$ months storage showed, that there was a significant decrease in the rate of formation of free fatty acids in the treated sample. According to Hoffpauir *et al.*[11] the free fatty acid content can be used as a practical screening index for seeding purposes. Since the percentage of germination decreases with increase of over 0.75 per cent free fatty acid content, it is suggested that this limit should form a basis for selecting the seed.

Insect and Fungus Infestation

Cottonseed when stored for long periods is prone to insect infestation particularly under unsatisfactory storage conditions. Storage places with many cracks in floors and in which seeds accumulate and remain unmoved from season to season provide breeding ground for an endemic population of insects. The one practical way to control this infestation is through fumigation. Among fumigants, methyl bromide is the one most widely used. In using this fumigant closed silos are desirable owing to the high toxicity of methyl bromide to human being. Suitable masks should be used when working in atmosphere containing methyl bromide.

Studies by Dudley and Neal[12] have shown that methyl bromide is safe to use as a commercial fumigant for most types of foods. Phillips and Bodenstein[13] fumigated large loads (450-550 tons) of cottonseed with methyl bromide at 3-6 lb./1,000 cu. ft. at the temperature range of 5°-18°C. for 24 hrs. in steel tanks provided with gas distribution arrangements. The gas kills pink boll-worm, flour-beetle adults and carpet-beetle larvae. Reeves[14] found that after fumigation with methyl bromide, 70-180 p.p.m. bromine is left in linters, hulls and meats. When the seed is hulled and the oil hydraulically pressed out, the cake is found to contain the entire bromine compound. Meuli *et al.*[15] found 30-50 per cent mixture of a zinc salt of 2.4.5-trichlorophenol combined with an inert diluent was effective as a fungicide for the treatment of cottonseed.

BIBLIOGRAPHY

1. Bailey, A. E., *Cottonseed and Cottonseed Products* (Interscience Publishers, Inc., New York), 1948
2. Condon, M. Z., Andrews, F. R., Lambou, M. G. & Altschul, A. M., *Science*, **105** (1947), 525
3. Karon, M. L., *J. Amer. Oil Chem. Soc.*, **24** (1947), 56-8
4. Jensen, E. A., Lambou, M. G., Andrews, F. R., Mayne, R. Y., Karon, M. L., Curet, M. A., Wilcox F.B. & Altschul, A. M., *J. Amer. Oil Chem. Soc.*, **28** (1951), 241-5
5. Whitten, M. E., *Oil Mill Gaz.*, **53** (3) (1948), 13-16
6. Lyman, C. M. Burda, E. J. & Olschner, P. Q, *J. Amer. Oil Chem. Soc.*, **25** (1948), 246-9
7. Altschul, A. M., Karon, M. L., Kyame, L. & Hall, C. M., *Plant Physiol.*, **21** (1946), 573
8. Jensen, E. A., Lambou, M. G. & Altschul, A. M., *Oil Mill Gaz.*, **54** (7) (1950), 43-6
9. Condon, M. Z., Lambou, M. G., Vignes, J. L., Loe., J. B. & Altschul, A. M., *Plant Physiol.*, **24** (1949), 241-54
10. Lambou, M. G., King, G. S. & Condon, M. Z., *Science*, **108** (1948), 563
11. Hoffpauir, C. L., Poe, S. E., Wiles, L. U. & Hicks, M., *J. Amer. Oil Chem. Soc.*, **27** (1950), 347-8
12. Dudley H. C. & Neal P. A., *Food Res.*, **7** (1942), 421-29
13. Phillips, G. L. & Bodenstein, W. G., *J. Econ. Ent.*, **41** (1948), 804-5
14. Reeves, W. A. & D'Aquin, E. L., *J. Amer. Oil Chem. Soc.*, **24** (1947), 217-19
15. Meuli, L. J., Thiegs, B. J. & Lynn, G. E., *Phytopathology*, **37** (1947), 474-80
16. Altschul, A. M., *Cott. Gin.* **52** (1951), 24-32
17. Altschul, A. M., *Oil Mill Gaz.*, **49** (1) (1944), 8-13
18. Karon, M. L. & Altschul, A. M., *Plant Physiol.*, **19** (1944), 310-325
19. Kulka, M., *Canad. J. Res.*, **B23** (1945), 106-110
20. Altschul, A. M., Karon, M. L., Kyame, L. & Caravella, M., *Oil & Soap*, **20** (1943), 258-262
21. Jensen, E. A., Lambou, J. S., Lambou, M. G. & Altschul, A. M., *Cott. Gin*, **53** (1952), 68-69, 84-85
22. *Methyl bromide — A Literature Review* (May & Baker Ltd., Dagenham, England), Jan. 1946

CHAPTER IV

PROCESSING OF COTTONSEED

The efficiency and overall economy of processing cottonseed in the United States of America have steadily improved as a result of continuous research and engineering development for the past fifty years. The first step in the processing of cottonseed consists in the separation of the kernel or meat from the whole seed. This includes seed cleaning, delinting and hull-separating operations (Fig. 1). Seed cleaning is generally accomplished in a pneumatic cleaner, especially when large quantities of impurities, like leaves, twigs, grits, etc. are present. The seeds are subsequently treated in a delinting machine. To the oil miller the linter is undesirable as it leads not only to oil losses due to imperfect separation of hull and meat but also diminishes the feed value of the cake due to excess of fibres.

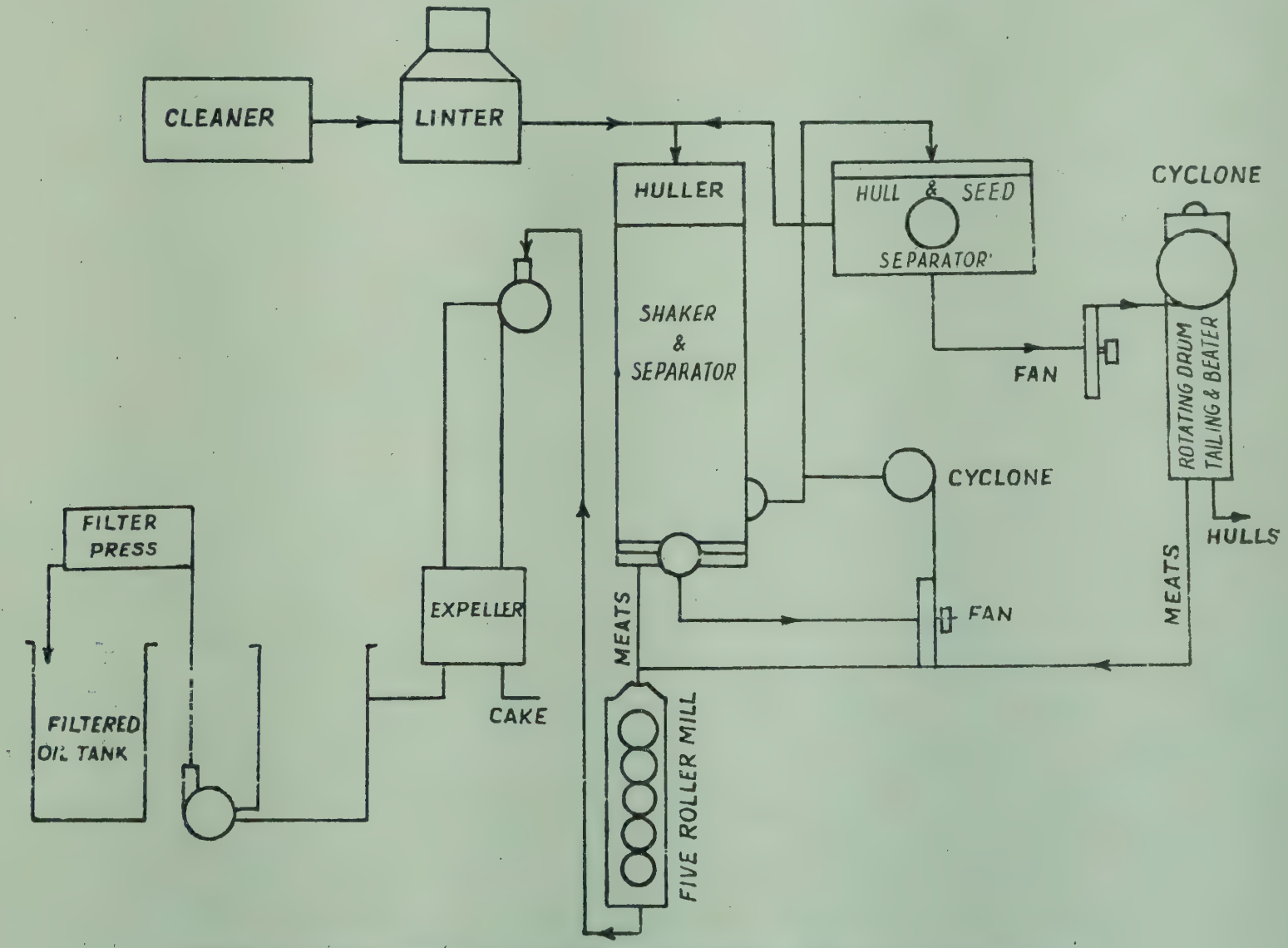

FIG. 1—PROCESSING OF COTTONSEED (FLOW DIAGRAM)

The delinting machine works on the same principle as the saw gin except that the saws have finer and closer-set teeth. The machine consists of a series of circular saws mounted on a horizontal revolving shaft and projecting through a set of steel ribs. During the operation, the saws revolve as the seeds fall on the ribs and cut the short fibres. These are subequently removed from the saws by large brushes rotating in the counter direction. Pneumatic conveyors carry these linters to the press room and compress them into bales. The delinting process is repeated in most mills for the second time and graded into "first cut" and "second cut" linters. In case the seeds are run through the machine only once, the linters are known as "mill run". Three kinds of delinting machines are employed in India. They are Continental, Carver, and Verner machines. A number of indigenous machines are also used for delinting.

The delinting operation is followed by hulling and separating processes. They vary greatly in detail and are classified into (1) double hulling, (2) single hulling and (3) universal hulling. In the double hulling process, delinted seeds after a prior treatment in a machine consisting of a huller, separator and beater, are passed for a second time in a similar but closely set machine to hull the remaining uncut seeds. In this process large amounts of fine hulls are formed which pass through the perforations of the shaker and are difficult to be separated from the kernels. Moreover, the entire quantity of hulls are exposed to oil absorption for a second time while hulling the uncut seed. In the single hulling process, the hulls and uncut seeds are therefore separated before they are treated in the second cut huller. Satisfactory results are usually obtained by close control of such factors as (a) moisture content of seed, (b) amount of linters left on seed, (c) degree of deterioration of seed, and (d) set up and operating conditions of separating machinery. Under satisfactory working condition the loss of oil due to absorption by hulls is less than 0.5 per cent. The method of universal hulling is often resorted to other methods when relatively low protein cake is required and a large amount of immature seeds and linters are present in seeds. It is a combination of single hulling and double hulling process, in which the hull fraction separated from the kernels in the single hulling process is passed through a closely set huller, shaker and beater for recovering the meats before going to storage. In all the hulling systems it is usual to allow 10-15 per cent hulls in the meats to control protein percentage in the cake.

After the hulling and separating processes, the meats are reduced to suitable flake thickness in a roller mill to promote efficient contact between the meats and moisture during cooking. A flaking thickness

of 0.005-0.01 in. is found to give optimum extraction results in commercial practice[6].

Mechanical Process of Oil Extraction

In the extraction of vegetable oils three methods are practised. They are hydraulic press, expeller and solvent extraction systems. Table 8 gives the average yield and percentage of cottonseed products obtained by hydraulic pressing of 1 ton of cottonseed[2].

TABLE 8—YIELD OF COTTONSEED PRODUCTS FROM ONE TON OF COTTONSEED BY HYDRAULIC PRESSING

	lb.	%
Cake	882	44.1
Hulls	471	23.5
Oil	315	15.8
Linters	191	9.6
Moisture and manufacturing loss	141	7.0
	2,000	100.0

Expellers are considered to be more advantageous than hydraulic presses. The following advantages have been recorded in the regular operation of a 100-ton per day cottonseed oil mill working with mechanical expellers in U.S.A.

(i) Increase of 11-12 lb. of oil per ton of seed;
(ii) less requirement of labour, as for instance, three operators replacing the normal 21 operators;
(iii) elimination of the cost of press cloth;
(iv) lower residual gossypol content in the meal; and
(v) higher gossypol content in oil improving stability to rancidity and low refining losses.

The expeller requires considerably more power. Most cottonseed crushing mills in U.S.A. have switched over to processing by expellers and, more recently, to expellers combined with solvent extraction systems. At present the modern expeller units are regarded all over the world as the standard for the mechanical extraction of oil and the hydraulic press is rapidly dropping from the world scene[7-9].

The steps in cottonseed oil milling are the same either by expeller or by hydraulic press systems, up to the flaking operation. The hydraulic press consists of a series of rectangular steel boxes placed one above the other. Each box is perforated at the bottom. The meats are placed in the boxes and hydraulic pressure is gradually increased to 4,000 lb./sq. in. The oil is squeezed from the meats through the

press cloth into an oil collector situated at the bottom of the press. In the hydraulic press the meats are usually kept under pressure from 20-30 minutes while in some mills the pressing time is prolonged in order to obtain more oil.

One of the leading American manufacturers of expellers, viz., Anderson, have improved expeller design to obtain more yield in oil. Table 9 gives the Anderson's models, their capacity and the residual oil content in cakes when whole and decorticated cottonseeds are treated.

TABLE 9—CAPACITY OF EXPELLERS AND RESIDUAL OIL CONTENT OF CAKE

Expeller model	Capacity (metric tons/24 hr.)		Residual oil in cake %
	Decorticated seed	Whole seed	
Anderson "Red Lion" model	6-8	5-7	5-7
„ "Duo" model	13-15	10-12	4-5
„ "Super Duo" model	22-25	16-18	3.5-4.5

The "Red Lion" model is the simplest design of a single pressing machine and contains a simple horizontal pressing barrel. A number of this type are operating in this country. Some of the continental machines imported into this country resemble this model in their main features. The Anderson's "Duo" expeller is the smallest expeller model having both vertical and horizontal pressing barrels and in its output is intermediate between the "Red Lion" and the "Super Duo" models. The largest in output is the "Super Duo" model and is claimed to be the most efficient continuous screw press available. The press is driven by two motors, one installed in the main gear case to drive the horizontal worm shaft and the second installed on the hopper feeder gear case to drive the vertical worm shaft. The use of motors results in a simpler, more practical and more effective machine particularly for handling large quantities of material.

The expeller treatment may vary somewhat according to the condition of the raw material but the general procedure is as follows: The flaked material from the "5-high Roller mill" is fed continuously into a separate steam drier or directly into the expeller-cooker or conditioner vessel by means of a variable feeder. It is cooked and dried to optimum moisture content for obtaining the best yields. The material then passes into the hopper feeder. It then enters the vertical barrel of the expeller which removes 20-50 per cent of the oil and passes continuously to the horizontal barrel, and finally, is discharged as a cake in a chip form, the oil having been recovered in the vertical and horizontal barrels. Since vegetable oils under

elevated pressure and temperature attack worm shafts of alloyed steel which are not hard faced, expeller designers have perfected steel compositions and case hardening formulae with high wear resistance[4].

Solvent Extraction

Many attempts have been made in U.S.A. since the beginning of this century to produce edible cottonseed products by solvent extraction on a commercial scale. The first commercial mill to process cottonseed by this process was Evans Process Works, Northern Indiana. This was followed by three other ventures between 1900 and 1919. They all failed to produce edible oil of acceptable quality and the meal was found to be toxic when fed to livestock. As a consequence, the solvent extraction process was not considered to be suitable for producing edible cottonseed products[10].

From 1945 research development on solvent extraction was intensified in U.S.A. Allis Chalmer's Manufacturing Company and Delta Products Company constructed a 200-ton per day continuous solvent extraction unit for cottonseed at Wilson, Arakansas (U.S.A.). A 350-ton continuous unit was erected at the Buckeye Cotton Oil Co. at Memphis., Tennesse for soyabean and cottonseed. Another 100-ton unit was erected by Struthes Wells Corporation and West Texas Oil Co. These three units marked the beginning of large-scale cottonseed oil extraction industry in U.S.A.[10] With the closing of the first season in 1948, over a lakh tons of cottonseed were processed by the three mills[11]. A year's operation showed that the control of gossypol required special attention over cooking practice. From the economical aspect, this solvent process showed 45 lb. more oil than hydraulic press operation with only four skilled men in the place of 32 men required for a 200-ton unit[12]. With proper control the oil colour and refining losses were comparable to hydraulic press operation. The extracted meal contained less residual oil and was a little brighter in colour and somewhat finer in texture; it was entirely satisfactory as cattle feed[11-13].

The first step in the extraction of cottonseed oil is treatment of raw material (press cake or kernel) in order to expose the oil cells to the action of solvent. This consists in cracking, heating and flaking. For solvent extraction systems good hulling and correct regulation of moisture content are necessary for trouble free operation. The presence of higher proportions of moisture darkens the colour of the meal due to the rupture of glands. With the optimum moisture content, the flakes become firmer and less subject to disintegration of "fines"[7,14]. It was found that a regulated moisture content of 9-9.5 per cent with a flake thickness of 0.011 in. gives the best results[6].

After flaking, the product is cooked at 165°F. or at a slightly higher temperature in a stack-cooker. The material then passes to the extractor where it comes into contact with the solvent and the oil is removed by washing and diffusion. The miscella or solvent-oil mixture obtained from the extractor is clarified by filtration and evaporated to remove the major part of the solvent and finally stripped under vacuum to remove the last traces of moisture and solvent in the distillation apparatus. The solvent vapours from the evaporator and stripper are condensed and water is removed by decantation before returning the solvent to the extractor. The extracted meal enters dryers where the last traces of solvent are removed by heat and recovered by condensation. The solvent-free meal is toasted, cooled, sifted and bagged in warehouses. The standard commercial unit can also handle other raw materials besides cotton-seed.

There are many different types of batch and continuous extractors described in the extensive literature published on the subject. In 1939, no fewer than 60 different extraction units were referred to in German, French, English and American patent and technical literature, but only a few had proved valuable in large-scale operation. The most popular types of extractors are those of Bollmann or Hansamuhle, Hildebrandt, Fauth, Miag, Wilhelm, Ford, Detrix, Bonotto, Allis Chalmers, Anderson, Kennedy and a few others[8].

Just as there have been various types of equipment, quite a variety of solvents have been used in the solvent extraction process. Among the solvents may be mentioned benzene, carbon disulphide, carbon tetrachloride, trichloroethylene, petroleum naphthas, alcohols, and so forth.

In the extraction of cottonseed oil by solvent extraction, pre-expellers combined with solvent extraction are gaining acceptance by cottonseed oil millers in U.S.A.[8,15]. Prior to World War II many European oil mills imported cottonseed from Egypt and India and processed it by a combination of expeller forepressing and solvent extraction. The pre-expellers used have the same mechanical features as other expellers with the difference in gearing, wormshaft and barrel bar spacings[8]. It is claimed that the present American pre-expeller model of "Super Duo" has three times the normal capacity with only one-third the power consumption, producing a cake of 8-10 per cent residual oil content[4]. It is also confirmed that the pre-pressing-solvent extraction system requires a little more power per ton of material and produces a meal with lower residual oil content when compared with direct-solvent extraction systems. As, for instance, in a typical cottonseed oil installation, the residual oil

in the meal was reduced to 0.3 per cent as compared with an average of 2.6 per cent for the previous season with direct solvent extraction[4].

Gastrock[9,16,17] and his associates have developed at the Southern Regional Research Laboratory, U.S.A., a new process for extracting oil from cottonseed, which overcomes the difficulties inherent in the conventional direct-solvent extraction and solvent extraction with pre-pressing. This process employs filtration as the major unit operation. It can handle cottonseed of a wide range of moisture contents. The process consists in mixing the cooked flakes with the solvent for 15-20 minutes in a mixing conveyor to form a slurry. The bulk of the miscella is separated from the slurry almost instantaneously by a 36 in. diam. continuous horizontal vacuum filter. The meal is later washed counter-currently with weaker miscellas and finally, with solvent. A miscella of high oil content (25-30 per cent) and an extracted cake of low residual oil and solvent contents were obtained. The "fines" are reduced to less than 0.3 per cent and the particles are coarser than those obtained by the direct solvent extraction method. The solvent required was less than one lb. per lb. of cottonseed meat. According to the inventors the new process cuts down capital cost of equipment and lowers operational expenses. Commercial tests on 18-ton per day pilot plant showed that the solvent extracted meal was superior in quality, with less than 1 per cent oil and 0.03 per cent gossypol contents and comparatively high protein solubility. The quality of oil was superior and the process was easily adoptable to other raw materials.

The Southern Regional Research Laboratory, U.S.A., developed a new and unique method of separating the pigment glands in cottonseed meat[8,18-20]. The meat is finely disintegrated in a slurry of inactive solvents, like commercial hexane and perchloro-ethylene, and allowed to settle. Due to the different specific gravities of the pigment gland and the meal, the glands float and the meals sink. The oil remains mixed with the solvent and is recovered by evaporation and distillation. The meal, free from pigment, is separated by filtration. A pilot plant devised on this principle and operating at this Laboratory produced 75 lb. of pigment gland and a ton of defatted cottonseed flour. The protein content of the meal is increased by about 60 per cent, since practically all of the gossypol group of pigments are removed in the form of intact glands; the meal and oil thus obtained are relatively light in colour and possess high biological value.

Some of the relevant literature on the subject are the following : Bonotto (*U.S. Pat,* 2,551,581) reduced the toxic gossypol in the solvent extracted meal to a nutritionally safe level of 0.05 per cent by cooking

at 216°F. for 30-60 minutes in presence of ammonia vapours (0.001-0.002% ammonium carbonate being added to the meal before cooking. Sulphur dioxide (0.00005-0.00002%) was found to be a good substitute for ammonia. Adamova and Lebedava[21] converted practically all free gossypol into the inert form by heating for 30-60 minutes at 100°C. with 25 per cent moisture content. Autoclaving at a temperature of 120°C. for one hour irrespective of the moisture content detoxified gossypol completely. Olcott[22] advocated detoxification of gossypol before hexane extraction by cooking. He found that extraction with ether and aromatic or chlorinated solvents removes gossypol and yields a meal of high biological value (since proteins and enzymes have not been broken down by heat). Milligan and Bird[23] found that by cooking above 200°F. the meal loses its nutritive value. Bendler and McNeil[14] with hexane and water as co-solvents ruptured the walls of pigment glands and extracted the toxic pigment along with the oil. Hutchins and Williamson (U.S. Pat. 2,484,831) treated cottonseed meats for a prolonged period with a solvent mixture consisting of methyl alcohol an aliphatic hydrocarbons below the denaturing temperature of protein and obtained an edible oil and meal; gossypol was converted to a brightly coloured insoluble non-toxic complex (bound gossypol). Graci *et. al.*[24] desolventized fine cottonseed meal in a meal dryer under controlled conditions. The free gossypol content was reduced by 69 per cent without affecting the protein solubility and the meal was granular in form. Eves *et al.*[25] found that the solvents in which water was more soluble gave deeply coloured oils with higher refining losses. Among the solvents (hexane, benzene, ethyl ether, acetone and butanone) hexane-extracted oils gave a light-coloured product on refining; acetone and butanone came next in order; others gave dark coloured oils. The solvents which yielded highly pigmented crude oils, produced meals lower in free gossypol content and higher in protein solubility. The use of solvents other than hexane for processing cottonseed depends on the development of more efficient methods of decolourizing oil. Improvement in meal quality is achieved at the expense of oil quality. D'Aquin *et al.*[26] found that hexane extracted oil refined and bleached at a temperature below 140°F compared favourably in quality and grade with oil obtained by hydraulic pressing. The test data given by Decossas[27] and his associates on hexane-extracted crude cottonseed oil and refined oil were as follows :

Crude cottonseed oil—Free fatty acids (oleic), 3.5%; sp.gr.$^{30^c}$, 0.913; refining loss, 12.5%; moisture, 0.093%;

Refined cottonseed oil—Iodine number, 105.5; colour 35 Y and 14.4 R; colour of bleached oil, 35 Y and 55 R.

Ayers and Scott[28] found that extraction with methylpentanes as compared with normal hexane resulted in an increased oil yield with lower refining losses and brighter colour during refining and bleaching. Regardless of the hydrocarbon used, Ayer and Dooley[29] observed that the cooking treatment prior to extraction improved the yield and colour of the oil. Based on solubility of water in isoproponal and acetone, Harris and Howard[30] pointed out that the miscibility of water with solvents was advantageous in extracting the oil.

BIBLIOGRAPHY

1. Bailey, A. E., *Cottonseed and Cottonseed Products* (Interscience Publishers, Inc., New York), 1948
2. *Cottonseed and its Products* (National Cottonseed Products Association, Inc., Memphis 3, Tenn.), 4 Edn, 1950
3. Jamieson, G. S., *Vegetable Fats and Oils* (Reinhold Publishing Corporation, New York), 2 Edn, 1943
4. *Extracting Vegetable Oils with Expellers Solvent,* Exsolex Process (Anderson & Co. V. D., Cleveland, 2 Ohio)
5. Turner, S. H., *Report on the Edible Vegetable Oil Resources of West Pakistan* (Lever Bros. & Unilever Limited, London), April 1949
6. Wamble, A. C., *Oil Mill Gaz.*, **53**(3) (1948), 31-4
7. Batson, D. M., Thurber, F. H. & Altschul, *J. Amer. Oil Chem. Soc.*, **28** (1951), 468-472
8. Markley, K. S., *Oil Mill Gaz.*, **51**(7) (1947), 27-31
9. Cole, L. U., *Oil Mill Gaz.*, **51**(7) (1947), 36-8
10. Mac Gee, A. E., *Oil Mill Gaz.*, **52**(2) (1947), 17-21
11. Bilbe, C. W., *Oil Mill Gaz.*, **53**(1) (1948), 39-41
12. Gordan, J., *Sth Pwr Ind.*, **65** (7) (1947), 7, 62-3, 73
13. Vix, H. L. E., Pollard, E. F., Spadaro, J. J. & Gastrock, E. A., *Industr. Engng Chem.* (*Industr.*), **38** (1946), 635
14. Bendler, A. J. & McNeil, D., *J. Amer. Oil Chem. Soc.*, **28** (1951), 164-170
15. Dunning, J. W. *Oil Mill Gaz.*, **56**(1) (1951), 64-68
16. Spadaro, J. J., Graci, A. V., Gardner, H. K., Parker, J. S., Laborde, E. J. & Gastrock, E. A., *Oil Mill Gaz.*, **56**(1) (1951), 77-81
17. Gastrock, E. A., D'Aquin, E. L. & Vix, H. L. E., *Cott Gin*, **53** (1952), 30-37
18. Vix, H. L. E., Spadaro, J. J., Westbrook, R. D., Crovetto, A. J., Pollard, E. F. & Gastrock, E. A., *J. Amer. Oil Chem. Soc.*, **24** (1947), 228-36
19. Spadaro, J. J., Persell, R. M. Murphey, Jr. C. H. Vix, H. L. E. McCourtney, E. J., Hecker, J. L. Pollrd, E. F. & Gastrock, E. A., 39*th Annual Meeting of Amer. Oil Chem. Soc.*, 1948
20. Boatner, C. H. & Hall, C. M., *Oil and Soap,* **23** (1946),123-8
21. Adamova, A. A. & Lebedeva, M. A., *Gigiena Sanit,* **12** (1947), 33-5
22. Olcott, H. S., *Industr. Engng Chem.* (*Industr.*), **33** (1941), 611-15
23. Milligan, J. L. & Bird, H. R., *Poult. Sci.*, **30** (1951), 651
24. Graci (Jr.), A. V., Gardner, H. K., Cucullu, A. F. Crovetto (Jr.), A. J., Spadaro, J. J., & Knoepfler, B. N., *J. Amer. Oil Chem. Soc.*, **29** (1952), 41-43
25. Eaves, P. H., Molaison, L. J., Black, C. L., Crovetto, A. J. & D'Aquin, E. L., *J. Amer. Oil Chem., Soc.*, **29** (1952), 88-94
26. D'Aquin, E. L., Spadaro, J. J., Vix, H. L. E., Pomenskii, J., Molaison, L. J. & Pollard, E F., *Oil Mill Gaz.*, **51** (10) (1947), 17-19

27. Decossas, K. M., Mackey, H. A. & Heughan, G. P., *Bureau of Agricultural and Industrial Chemistry; Agricultural Research Administration, United States Department of Agriculture* (A.I.C. 257), March 1950
28. Ayers, A. L. & Scott, C. R., *J. Amer. Oil Chem. Soc.*, **28** (1951), 348-51
29. Ayers, A. L. & Dooley, J. J., *J. Amer. Oil Chem. Soc.*, **25** (1948), 372-9
30. Harris, W. D. & Howard, J. W., *J. Amer. Oil Chem. Soc.*, **27** (1950), 273
31. Goss, W. H. *Oil Mill Gaz.*, 53 (3) (1948), 16-21
32. Gastrock, E. A. & D'Aquin, E. L. *Oil Mill Gaz.*, **53** (4) (1948), 13-21
33. Bonotto, *M., Oil and Soap*, **23** (1946), 297
34. Krishnamurthi, M. N., *J. Sci. Industr. Res.*, **9A** (1950), 17
35. Tray, S. E. & Bilbe, C. W., *Chem. Engng*, **54** (1947), 139
36. Knoepfler, B. N. Vix, H. L. E. & Thurber, F. H., *Cott. Gin*, **53** (1952), 61-66
37. Falkenburg, A. J., *Oil Mill Gaz.*, **56** (1951), 36
38. Gastrock, E. A., *Oil Mill Gaz.*, **56** (1951), 35-37

CHAPTER V

NUTRITIONAL ASPECTS OF COTTONSEED PRODUCTS

Cottonseed has been used in livestock feeds for many years in India, although its cake or meal is superior in nutritive value. About two decades ago, a few cottonseed oil mills were established in parts of Sind. Since then the cottonseed crushing industry made little progress on account of the low price offered for the cake as it found little favour with the farmer for feeding cattle.

Cottonseed meal has been extensively used in U.S.A. as a source of protein in livestock feeds. Occasionally, delinted and undecorticated whole cottonseed is crushed in expeller mills and the meal is sold on 28 per cent protein basis. This meal had a fairly good market in comparison with cottonseed meal containing 43 per cent protein obtained by crushing decorticated meats and provides a more remunerative outlet for the hulls.

Feeding trials at the Texas Agricultural Experiment Station, U.S.A. showed that animals gained in weight when cottonseed was supplemented to the normal feeds in limited quantities of ½ lb. per 100 lb. body weight[3]; when fed in larger amount cattle went off the feed. In another feeding trial the addition of molasses to cottonseed meal and hulls improved the palatability and raised the consumption. Comparative feeding values of American and Indian varieties of cottonseed and their cakes were determined at the Agricultural Research Institute, Lyallpur. In one experiment Lander and Dharmani[4] substituted the fuzzy American type with deshi seed and found no harmful or adverse effect either on cattle or on the yield and quality of milk. Table 10 gives the starch equivalent and digestible protein content of different varieties of cottonseed and cake[5].

Based on these trials Lander and Dharmani concluded that 8 per cent fat is the maximum limit that could be satisfactorily assimilated by cattle. The figures for starch equivalent and digestible protein showed the nutrients were better utilized by the animals in the form of cake than in the form of seed[5].

TABLE 10—STARCH EQUIVALENT AND DIGESTIBLE PROTEINS OF COTTONSEED AND CAKE

	Per 100 lb. of seed or cake	
	Starch equivalent (lb.)	Digestible proteins (lb.)
Fuzzy American cottonseed (289F./43)	85.95	14.0
„ „ „ (289F.)	68.25	11.5
Naked American cottonseed (4F.)	62.85	10.67
Deshi cottonseed	69.57	8.00
Undecorticated cottonseed cake	60.56	17.97
Decorticated cottonseed cake	59.36	29.10

The Indian Council of Agricultural Research has investigated the relative nutritive value of cottonseed and its cake and the data[6] are provided in Table 11.

Cattle fed exclusively on cottonseed and green fodder provided ghee which had a higher refractive index and iodine value, lower Reichert-Meissl and Polenske values and greater stability to oxidative spoilage than ghee obtained from cattle fed on straight green fodder ration. The absorption and secretion of carotene and vitamin A in butter fat are poor when the ration is predominently cottonseed[7]. Hulls of Indian cottonseed contained 40-60 per cent total digestible nutrient and 0.38 per cent digestible protein[8]. Cattle could eat 20 lb. hulls per day and hulls were preferred to wheat straw. Hussain *et al.*[8] recommended supplementing the hulls with silage, legume hay or with good quality green fodder, or with calcium and phosphorus in mineral form since cottonseed hulls are low in calcium and phosphorus.

However, there are several minor uses of the meal among which the possibility of utilizing cottonseed meal on a large-scale as a source of food for human beings is at present of considerable interest. The cottonseed flour obtained from a specially processed cake constitutes a large potential source of high proteins, certain vitamins and essential amino acids and is practically free from starch. From a ton of cottonseed of average quality that would produce 950 lb. of 43 per cent protein meal, about 300 lb. of edible cottonseed flour is obtained. The flour has a relatively light colour and a rather pleasant and a characteristic nutty flavour. A typical analysis is given in Table 12.

Dr. D. Breese Jones[10,11] gives the following official comment on the possibilities of cottonseed flour in foods : "The high protein content of cottonseed flour is exceeded by only a few other foods. The digestibility of the total protein of cottonsed flour is as good as that of peas 80 per cent that of meat and 90 per cent that of cereals. The

TABLE 11—AVERAGE PERCENTAGE COMPOSITION, DIGESTIBILITY COEFFICIENTS AND NUTRITIVE VALUE OF INDIAN COTTONSEED AND CAKE

	Av. percentage composition		Av. digestibility coefficients		Nutritive value (lb.)	
	Seed	Cake	Seed	Cake	Seed	Cake
Total ash	4.66	6.50				
Fibre (carbohydrates)	25.74	24.11	63.00	74.00	34.65*	39.96*
Soluable carbohydrates (nitrogen-free extract)	30.98	37.40	59.00	59.00		
Ether extract (fat)	20.60	9.15	90.00	98.00	18.50*	8.00*
Crude protein	18.02	22.84	69.00	85.00	12.49*	19.42*
Digestible crude protein					11.24†	17.48†
Starch equivalent					68.40†	59.30†
Total digestible nutrients					80.00†	71.60†
Nutritive ratio					6.10	3.10

Ether extract—includes all the soluble substances of a feed including true fats, fatty acids and such other substances soluble in ether.

Crude protein—includes the true protein and non-protein nitrogenous compounds.

Digestible crude protein—The available protein fraction in a feed.

Starch equivalent—The available energy in a feeding-stuff as usually reported in the U.K. and some other European countries

Total digestible nutrients—The energy value of a feed as generally reported in the U.S.A.

Nutritive ratio—Nutritive ratio is the ratio of digestible protein to the digestible non-nitrogenous nutrients in a feed. The amount of non-nitrogenous nutrients is reckoned as the sum of digestible total carbohydrates and digestible ether extract multiplied by 2.25. The value of nutritive ratio gives the idea of the proportion of digestible protein in relation to other nutrients. The ratio is narrow in the case of protein-rich feeding stuffs and wide where the feeds are rich in carbohydrate or in fat or in both.

* Digestible nutrient per 100 lb. dry material

† Digestible nutrient per 100 lb. raw material

TABLE 12—COMPOSITION OF COTTONSEED FLOUR[9]

Proximate analysis	%	Mineral	%	Vitamin	µg/g.
Moisture	6.34	Phosphorus	1.26	Thiamine	10.4
Protein	57.53	Calcium	0.20	Riboflavin	10.2
Fat	6.45	Magnesium	0.65	Niacin	84.0
N-free extract	21.38	Iron	0.012	Pantothenic acid	25.5
Fibre	2.06				
Ash	6.24				

protein of cottonseed flour is a good source of the nutritionally essential amino acids. It has a growth promoting value approximately 4.5 times that of wheat flour. It is well suited to supplement the proteins of certain foods, particularly, those of cereal grains chiefly wheat, as wheat flour is known to be deficient in some of the essential amino acids, abundant in cottonseed flour. Addition of as little as 5 parts of cottonseed flour to 95 parts of wheat flour produces a mixture containing 16 per cent more protein than wheat flour alone, and a protein combinaton definitely superior in its growth promoting

value to the same quality of proteins from wheat flour." Table 13 gives an analysis of cottonseed meal by microbiological procedure for essential amino acids[1,12].

TABLE 13—AMINO ACID CONTENT OF COTTONSEED MEALS

	Solvent extracted (7.75% N)	Hydraulic pressed (6.29% N)
Arginine	5.18	4.13
Histidine	1.25	1.04
Lysine	2.25	1.60
Valine	2.38	1.92
Leucine	2.78	2.38
Isoleucine	1.90	1.60
Methionine	0.71	0.56
Phenylalanine	2.51	2.00
Threonine	1.63	1.33
Tryptophane	0.75	0.61

Byadagyan *et al.*[13] pointed out that in the content of some amino acids cottonseed closely resembles animal proteins. They conducted human feeding experiments on the biological value of cottonseed flour by using 10 per cent cottonseed flour with 90 per cent rye flour in the form of bread. At the end of one year of experimentation, no ill-effects of any kind was found in any of the subjects. Chemical and biological tests did not show any abnormalities or pathological changes in the epithelial lining of internal organs. It was concluded that cottonseed flour is non-toxic for human beings if consumed in small amounts.

Cottonseed flour is finding increased use in a variety of baked products. It is free from any tendency towards flavour reversion and is stable towards oxidative rancidity. Biologically, it is a concentrated source of protein, minerals, and vitamins of the B group. It imparts tenderness and shortness in baked products. Tondeur[14] produced a flour from pressed cakes which can be used in pastry mixes up to $12\frac{1}{2}$ per cent. With improvement in cooking conditions in oil industry, the pressed cakes produced a better grade flour which could be used in amounts up to 20 per cent in mixes. The solvent extracted cottonseed cake produced a high grade flour free from odour and colour. Andrea and Overman[15] substituted soya and cottonseed flours in pastries and found no effect on their breaking strength. Pastries containing 10 per cent soya flour and 3-10 per cent cottonseed flour were good in colour and texture and exerted anti-oxidant action in raw mixes. Kuppuswamy *et al.*[16], on substituting cottonseed and sesame flours in South Indian rice diet, found marked improvement in growth in rats.

The pigment glands of cottonseed are mechanically strong and resistant to the action of most solvents. They have deleterious physiological effects if present in high proportions in cottonseed meal. Pigment gland-free cottonseed meal prepared by the floatation process is free of toxicity and can serve as a protein supplement in poultry ration[17,18]. Lillie and Bird[19] reported that the extent of growth depression on chicks was directly proportional to the gossypol intake. Ambrose and Robbins[20] observed that diets containing 32 per cent gland-free cottonseed meal containing 0.05 per cent gossypol exhibited no sign of toxicity, while meal containing 1.10 per cent gossypol when fed at 15 per cent level in the diet definitely suppressed growth. Eagle and Bialek[21] noted by experiments on rats that the efficiency of cottonseed pigment glands as growth inhibitors was closely correlated with gossypol content.

Cottonseed oil is extensively used as edible fat in U.S.A. It is used in the manufacture of shortening, margarine, salad dressing, mayonnaise, cooking and salad oils. It supplies about 270 calories per ounce compared with 115 calories per ounce furnished by proteins and carbohydrates. There are conflicting reports in the literature as to whether various animal and vegetable fats have similar nutritive properties aside from their vitamin content. But, it is known that with the exception of a few fats having abnormally high melting points all are equally well absorbed in the human system, the coefficient of digestibility approaching 95 per cent[22]. The office of Home. Economics of the United States Department of Agriculture made a thorough study of the digestibility of fats in man over a period of years. The data presented by Longworthy represents results on 34 vegetable and 19 animal fats and oils. The more important of the results are reproduced in Table 14.

Cottonseed oil, like other vegetable oils, excels animal fats in the proportion of essential fatty acids, varying from 50-75 per cent and is therefore effective in the treatment of fat-deficiency diseases. The oil after processing keeps for long periods even at room temperatures. It is especially suitable for frying because of its high smoking temperature[9].

TABLE 14—COEFFICIENTS OF DIGESTIBILITY OF IMPORTANT OILS AND FATS[9]

	Coefficient of digestibility (av.)
Butter	97.0, 88.9
Coconut	97.9, 88.7
Cottonseed	97.6, 96.9
Peanut	98.3
Rapeseed	98.8
Sesame	98.0

BIBLIOGRAPHY

1. Guthrie, J. D., Hoffpaur, C. L., Stansbury, M. F. & Reeves, W. A., *Survey of the Chemical Composition of Cotton Fibres, Cottonseed, Peanuts and Sweet Potatoes—A literature Review*, United States Department of Agriculture. Agricultural Research Administration, Bureau of Agricultural and Industrial Chemistry, 1949
2. Turner, S. H., *Report on the Edible Vegetable Oil Resources of West Pakistan* (Lever Bros. & Uniliver Ltd., London)
3. *Tex. agric expt. Sta. Bull.* 724, Sept. 1950
4. Lander, P. E. & Dharmani, L. C., *Ind. J. vet. Sci.*, **15** (1945), 22-7.
5. Mohammad Afzal, *Indian Cott. Gr. Rev.*, **2** (1948), 166-177; **6** (1952), 45
6. Sen, K. C., *The Nutritive values of Indian Cattle Foods and Feeding of Animals, Indian Council of Agricultural Research Bull.* 25, 3 Edn, 1952
7. Patel, B. M. & Ray, S. C., *Indian J. Dairy Sci.*, **2** (1949), 30-8; 146-53
8. Hussain, A., Halini, A., & Wahhab, A., *J. Agric. Sci.*, **41** (1951), 379
9. Bailey, A. E., *Cottonseed and Cottonseed Products* (Interscience Publishers, Inc., New York), 1948
10. Jones, D. B. & Divine, J. P., *J. Nutr.*, **28** (1944), 41-49
11. McMath, C. W., *Proc. Cott. Res. Congr. Dallas*, 1945, 22-25
12. Lyman, C. M., Kuiken, K. & Hale, F., *Tex. agr. expt. Sta. Bull.* 12, 692
13. Bydagyan, F. E., Vladimirov, B. D. & Levirskii, L. M. & Suchenov, K. A., *Gigiena Sanit* (1947), *Congs Belge*, 28-33
14. Tondeur, G., *Bull. agric.* (7), **38** (1947), 1, 3-58
15. Overman, A., *Food Res.*, **16** (1951), 39-42
16. Kuppuswamy, S., Giri, K. V. & Subrahmanyan, V., *Sci. Cult.*, **12** (1946), 249-50
17. Groschke, A. C., Rubin, M. & Bird, H. R., *Poult. Sci.*, **26** (1947), 310.
18. Boatner, C. H., *Poult. Sci.*, **27** (1948), 315
19. Lillie, R. J., & Bird, H. R., *Poult. Sci.*, **29** (1950), 390
20. Ambrose, A. M., & Robbins, D. J., *J. Nutr.*, **43** (1951), 357
21. Eagle, E., & Bialek, H. F., *Food Res.*, **15** (1950), 232
22. Holmas, A. D. & Dauel, Jr., H. J., *Amer. J. Physiol.*, **54** (1921), 479
23. Massey, Z. A., Boggess, Jr., T. S. & Holley, K. T., *Brochure on Georgia Experiment Station Circular* 138—*Composition of Protein Supplement*, July 1942

CHAPTER VI

REFINING OF COTTONSEED OIL

Crude cottonseed oil as extracted from the seeds is not quite suitable for edible purposes and requires to be refined. The commercial refining process consists of (1) the removal of gums phosphatides and free fatty acids by neutralization, (2) bleaching and (3) deodorization. In principle, these operations are relatively simple, but in practice a thorough knowledge of operating conditions is essential to secure finished products of high quality.

Degumming

Prior to the actual neutralization of free fatty acids by alkali, a treatment to remove gums is very necessary. When cottonseed oil contains 1 per cent free fatty acids and 2 per cent phosphatides and other non-glyceride impurities the refining loss in the common batch method may be as high as 7-8 per cent. For removing gums, phosphatides and other mucilage Howard *et al.* (*U.S. Pat.*, 2,416,146) mixed crude cottonseed oil intimately with 2 per cent water and subjected the mixture to continuous centrifugal separation at 135°F. The oil was then recovered with two volumes of furfuraldehyde (saturated with water) at 110°F. by continuous counter-current operation. The extracted oil was then washed with 10 per cent of a 2 per cent solution of caustic soda and the foots removed at 180°F. by continuous centrifugal separation. The resultant oil having a colour of 35 yellow and 4.5 red showed 2.4 per cent refining loss, as compared with 35 yellow, 16.6 red and 3.5 per cent refining loss when refined without initial degumming. Another process of degumming consisted in precipitating the undesirable substances by the addition of sodium silicate or phosphoric acid and alum and heating with live stream.

Neutralization

In the batch system of oil refining, the crude oil is pumped to the neutralizer. It is then heated to 75-80°F. with slow agitation. Alkali pre-heated to 212°F. is sprayed uniformly over the oil. The

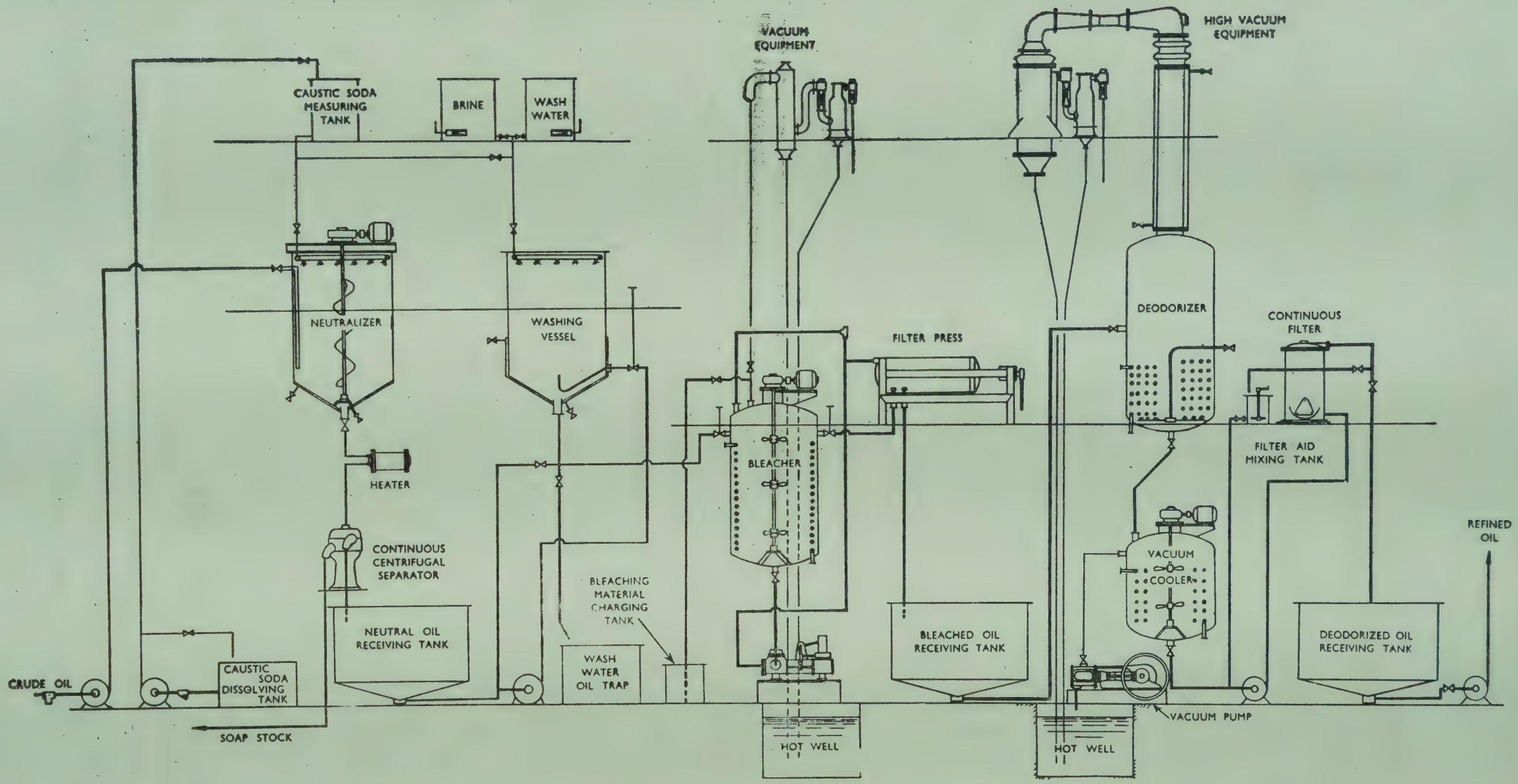

FIG. 2—REFINING PLANT (FLOW DIAGRAM)

amount and strength of caustic soda desired is determined by standard laboratory tests. Table 15 shows the percentage of lye required to neutralize the free fatty acids in oils of varying acidity[2].

TABLE 15—PERCENTAGE OF LYE REQUIRED TO NEUTRALIZE FREE FATTY ACIDS IN OIL

Free fatty acids %	percentage lye (20° Be.)
1.0	0.99
1.5	1.49
2.0	1.98
2.5	2.47
3.0	2.97
3.5	3.46
4.0	3.95
4.5	4.45
5.0	4.94

In commercial refining process, the removal of colour from crude cottonseed oil is directly related to the concentration of caustic lye, length of time and speed of agitation and indirectly to the temperature of the oil during agitation with sodium hydroxide solution. Concentrated alkali of 20°-30°Be. has powerful degumming action; it also improves the colour as the soap stock formed is easily separated from the oil. But it causes excessive hydrolysis. It is perferable to select weaker lyes for refining oils with a low free fatty acid value and more concentrated lyes for oils with high free fatty acid content. To produce a bright coloured oil, excess of caustic soda is necessary. For oils containing 2 per cent or less of free fatty acid an excess of 0.2-0.35 per cent dry sodium hydroxide may be used. For oils with fatty acids contents above this range, the required excess will be higher and in certain cases may be as much as 1 per cent. During neutralization, the temperature of the alkali must be a little higher than that of the oil for preventing the formation of emulsions. The mode and speed of stirring are also important factors in emulsification and salting out operations. Efficient results are obtained by stirring at slow speed just prior to the addition of lye. After the addition of caustic lye, agitation at high speed is necessary for 15-20 minutes, the temperature of oil being slowly raised to 125-135°F.

McGaskell (*U.S. Pat.*, 2,459,082) has described an improved rotary alternating pressure and vacuum filter assembly, especially suited for separating refined cottonseed oil from foots. Sharma and Lal[6] found that 9.27 per cent caustic soda effected the best removal

of colour at 30° and at 40°C. in single-stage refining. Bhasin and Aggarwal[7] produced bright coloured oil from dark crude cottonseed oil of acid value 5.8 by re-refining with alkali of 15 per cent strength, with 0.5 per cent excess. The speed of agitation was increased from 150-200 to 400-500 r.p.m. Kroonen and Feuge[8] showed that the colour of the product was improved by increasing caustic soda concentration, rate of agitation and shear in mixing oil and alkali. About 0.2 per cent caustic soda (on weight of oil) used in the form of 14-24 per cent solution, 5-10 minutes agitation at a maximum temperature of 65°C. produced maximum reduction in colour. Cavanagh[9] recommended 20° Be. strength of caustic soda and temperature below 85°F. for efficient colour removal and minimum oil loss. Further, he suggested more vigorous agitation in the case of dark oils. Folzenlogen[10] (*U.S. Pat.*, 2,563,327 and 2,563,328) improved the refining yield of crude cottonseed oil by the addition of a non-ionic compound to the oil before the addition of caustic lye. This compound may be either a poly-hydroxy alcohol (containing up to 3 hydroxy groups and 2-8 carbon atoms) or a non-ionic organic derivative of either in which at least one unreacted hydroxyl group has been substituted. Willylange (*U.S. Pat.*, 2,551,496) reduced neutral oil losses in alkali refining to 25 per cent by mixing salicylic acid (about 0.01%) with the crude oil, agitating for 5 minutes, followed by the addition of about 1 per cent water with continued agitation for another 15 minutes. Alkali refining is then carried out in the usual manner. Edward (*U.S. Pat.*, 2,415,140) treated 60,000 lb. charge of oil containing 1.6 per cent free fatty acid with 1,871 lb. caustic soda of 12.9 per cent strength containing 750 lb. crude glycerol. The refining loss was 3.7 per cent in contrast to 5.8 per cent when processed according to the prior method. Similar results in oil savings were obtained in continuous refining trials. In batch refining, the neutralizers are suitably dimensioned for one batch for 24 hr. to allow sufficient settling time to ensure minimum refining losses.

Neutralization with soda ash has also been attempted. It was found that soda ash has a much less powerful saponifying action than caustic soda and processing difficulties are increased due to the formation of carbon dioxide during neutralization of the free acid in the oil.

Bleaching

After settling, the bright neutral oil in the neutralizer is drawn off through a swinging suction line to the bleacher. The oil is washed by spraying with 10-15 per cent hot water (180°F.) or with dilute brine to facilitate breaking of emulsion and to remove traces of caustic soda or soap stock entrained in the oil. Finally, it is

dried in vacuum (2-3 in. mercury). For decolourizing coloured oil it is better to use first grade activated carbon in conjunction with pulverized fuller's earth, or bleaching clay. The ratio of activated carbon to clay will vary according to the character of the oil to be treated, and the kind of clay used. It was found that such a mixture of 90-95 parts clay and 5-10 parts of carbon reduces the total bleach requirements by 30-50 per cent. The oil is heated under vacuum to a temperature slightly in excess of 212°F. A mixture of bleaching clay and activated carbon is added and high speed agitation continued for 20-45 minutes. Agitation is continued until test samples show that the maximum effect has been produced. The treated oil is pumped from the bleacher through a filter press and the clear oil run to the storage tank for further treatment. The oil remaining in the cake can be partly recovered by steaming or more completely by extraction with organic solvents. Feuge and Jenssen[11] demonstrated that bleaching of cottonseed oil in hexane with clay-carbon mixture brought about greater reduction in colour and improved the efficiency of the utilization of clay with respect to oil loses. According to the claims of Sanders (*U.S. Pat.*, 2,555,098) bleaching of oils before, rather than after, drying removes colour more effectively.

Deodorization

The deodorization process is omitted when the oil is refined for industrial applications, but it is an essential step for edible oils. Hydrogenated oils have to be deodorized to remove the hardening flavour. The bleached oil is drawn into the deodorizer by vacuum and the temperature raised to 176-194°F. by an internal heating coil. Super-heated steam is blown in and during the treatment which may last for 4-6 hr., the temperature of the oil is maintained at 350-425°F. A high vacuum (0.25 in.) is maintained to reduce the consumption of steam and cooling water and to minimise the duration of high temperature. Heat economy in the deodorization process is an important factor. Savings in heat are obtained by the use of "Downtherm" system in which use is made of a high boiling substance (e.g., diphenyl ether) as the heat transfer medium. The oil is let into a vacuum cooler equipped with a high speed agitator and cooled under exclusion of atmospheric oxygen. Hickmann (U.S. Pat. 600,403) produced bland deodorized fats of enhanced stability by subjecting cottonseed oil to high vacuum distillation (< 10 mm.; 80-280°C.) and steam treatment (6-12 mm.; 175-225°C.).

BIBLIOGRAPHY

1. Bailey, A. E., *Cottonseed and Cottonseed Products* (Interscience Publishers, Inc., New York), 1948
2. Bailey, A. E., *Industrial Oil and Fat Products* (Interscience Publishers Inc., New York), 1951
3. Jamieson, G. S., *Vegetable Fats and Oils* (Reinhold Publishing Corporation, New York), 2 Edn, 1943
4. Watermann, H. I., *Hydrogenation of Fatty Oils* (Elsevier Publishing Co., New York), 1951
5. *Literature Review on Oils and Fats* (Council of Scientific & Industrial Research, New Delhi), 1949, 1950 & 1951
6. Sharma, A. P. & Lal, J. B., *Proc. Annu. Convention Oil Technol. Assoc. India,* **2** (1945), 30
7. Bhasin, M. M. & Aggarwal, J. S., *J. sci. indust. Res.,* **12A** (1953), 193
8. Kronen, J. G. & Feuge, R. O., *J. Amer. Oil Chem. Soc.,* **29** (1952), 65-71
9. Cavangh, G. C., *J. Amer. Oil Chem. Soc.,* **28** (1951), 377
10. Folzenlogen, R. G., *J. Amer. Chem. Soc.,* **28** (1951), 448
11. Feuge, R. O. & Jenssen, H. J., *J. Amer. Oil Chem. Soc.,* **28** (1951), 429-32

CHAPTER VII

EXAMINATION OF COTTONSEED AND ITS EDIBLE PRODUCTS

Despite the considerable progress made in the development of analytical methods, the industries are not familiar with general procedures for sampling and analysing oils and oilseeds. Standard procedures for the commercial evaluation of oils and oilseeds have been evolved by the American Oil Chemists' Society and adopted as standards in U.S.A. The methods are revised at regular intervals. In the interest of standard procedures among cottonseed oil millers in the country some 15 methods adopted by the American Oil Chemists' Society are given below. A list of apparatus and reagents required is given in Appendix A.

1. Sampling

A.O.C.S. Official Method Aa 1-38

Procedure—Make an imaginary division of the car load into 4 square sections. In the centre of each, dig a hole 30 in. deep. Take about 15 lb. of seed from the sides and bottom of each hole and place in a suitable moisture-proof bag, closing same to prevent changes in moisture. The sample representing the entire car shall be not less than 50 lb.

2. Foreign Matter in Cottonseed

A.O.C.S. Official Method Aa 2-38

Procedure—(a) Weigh a sample of about 1000 g. Pass the sample over the 6-mesh screen. Remove as much foreign matter as possible and pick out the remainder by hand after spreading out on a clean dry surface.

(b) Place the cleaned sample in any efficient mixer and mix by revolving 10 times at the rate of 5 r.p.m. Empty the sample into a large piece of paper and divide into 4 quarters with a large spatula, but avoid mixing.

(c) Return quarters 1 and 3 to the original sample containers. Place quarters 2 and 4 in suitable containers with tight-fitting covers for the determination of moisture, oil, nitrogen and free fatty acids.

$$\text{Foreign matter (\%)} = \frac{\text{Weight of foreign matter} \times 100}{\text{Weight of sample}}$$

3. Moisture and Volatile Matter of Cottonseed, Meats and Cake

A.O.C.S. Official Method Aa 3-38, Ba 2-38

Scope—Excess moisture in cottonseed is due primarily to weather conditions but frequently is also due to improper handling at the producing centres; it tends to heat and deteriorate in quality and quantity particularly when the moisture is over 12 per cent.

Procedure—Weigh accurately as rapidly as possible duplicate samples of 5 to 10 g. each into tared moisture dishes. Slip the cover on the bottom of the dish and place the uncovered dish in the oven and dry at 101°±1°C. for 12-16 hr. for seeds and 2 hr. for meats and cake. Remove the dishes from the oven, cover immediately, cool in a desiccator to room temperature and weight.

$$\text{Moisture and volatile matter (\%)} = \frac{\text{Loss in weight} \times 100}{\text{Weight of sample}}$$

4. Oil Content in Cottonseed, Cake, Meal and Hull

A.O.C.S. Official Method Aa 4-38, Ba 3-38

A. *Preparation of sample from cottonseed*—(a) The following preliminary treatment of cottonseed is essential before the determinations of oil, nitrogen, and residual lint. It is essential that each step in the analysis of samples of cottonseed be executed promptly with a minimum exposure to oxidation. Once started, analytical operations should proceed continuously without interruption or delay.

(b) Dry about 60 g. portion of mixed sample for 2 hr. at 130° ± 3°C. Absorb into inner walls and bottom of fuming vessel (unglazed porus earthenware) 1.5 ml. HCl (sq. gr., 1.19). Acid should be well distributed over inner sides and bottom of vessel and should be completely absorbed so that no drops remain.

(c) Place the dried seed in the prepared vessel, cover with a watch glass or clay lid, place the vessel in the fuming oven and heat for one hour. Increase the oven temperature gradually but do not exceed 115°C. The lint should become loose and brittle with this treatment but not scorched.

(d) Adjust the Bauer Mill so that it will grind the treated seed to a fine meal. Grind the entire fumed sample. Open the mill and carefully brush all remaining ground seed on to a smooth surface and add to the main portion.

CAUTION: It is important that there is a minimum loss in grinding. If more than 1 g. is lost, the sample is invalid and should be discarded.

(e) Mix the sample thoroughly. This may be done by placing the entire ground sample into a one-half gallon Mason fruit Jar containing a large rubber stopper. Replace the cover and shake vigorously until thoroughly mixed. Transfer the contents to an air tight container. This sample is used for the determination of oil, nitrogen and second (ground) moisture. Weighings for all determinations should be made at the same time.

B. *Procedure*—(a) Weigh accurately 4 to 5 g. of the ground sample into a filter paper and enclose in a second filter paper folded in such a fashion as to prevent escape of the meal. The second paper is left open at the top like a thimble. A piece of absorbent cotton may be placed in the top of the thimble to distribute the solvent as it drops on the sample.

(b) Place wrapped sample in the Butt extraction tube and assemble the apparatus. Add about 25 ml. of petrol-ether into the tared extraction flask before attaching to the tube.

(c) Heat on a water bath or electric hot plate at such a rate that the solvent will drop from the condenser on the centre of the thimble at the rate of at least 150 drops per minute.

(d) Keep the volume of solvent fairly constant by adding enough to make up for any that may be lost due to evaporation. Continue extraction for 4 hrs.

(e) Cool and disconnect the extraction flask. Evaporate the ether on a steam or water bath until no odour of ether remains. A gentle stream of clean dry air may be used to facilitate removal of the solvent. Cool to room temperature, carefully remove any moisture or dirt from the outside of the flask and weigh. Repeat heating until constant weight is obtained.

(f) Determine the moisture in the ground sample as directed in A.O.C.S. Official Method Aa 3-38, Ba 2-38.

C. *Calculation*

$$\text{Oil in ground sample } (\%) = \frac{\text{Weight of oil} \times 100}{\text{Weight of sample}}$$

$$\text{Oil, desired moisture basis (\%)} = \frac{\text{F (100-\% moisture desired)}}{\text{100-\% moisture in ground sample}}$$

F = % oil determined in ground sample.

5. Protein Content in Cottonseed Cake and Meal

A.O.C.S. Official Method Aa 5-38, Ba 4-38

A. *Scope*—Since cottonseed and its meal has been a staple food, its nutritive value is based on protein content which may be expressed in its equivalent ammonia or nitrogen percentages.

B. *Procedure*—(a) Use a sample prepared as directed in A.O.C.S. Official Method Aa 4-38, section A, paragraph (e).

(b) Weigh 1.7032 g. of sample into Kjeldahl flask. Add about 0.5 g. of mercury (0.7 g. of mercuric oxide), 10 g. of potassium or sodium sulphate and 25 ml. sulphuric acid.

(c) Place the flask on the digestion rack (in an inclined position) and heat, below the boiling point of the acid, for 5 to 15 minutes, or until frothing ceases.

(d) Increase the temperature and digest completely. A good indication of this is when the liquid becomes clear and colourless, but to be certain, heating should be continued for at least 30 minutes beyond this point.

(e) Cool, add about 300 ml. of water, a few granules of zinc to prevent bumping, and sufficient sulphide solution (usually about 25 ml.) to precipitate all of the mercury.

(f) Transfer accurately a sufficient quantity of the standard acid into receiving flask so that there will be an excess of at least 0.5 ml. of 0.5 N acid. Add sufficient distilled water to cover the end of the outlet tube and attach to outlet end of condenser tube. The distillate should discharge through a glass tube at the bottom of the receiving flask.

(g) Mix thoroughly and add sufficient alkali solution (usually 60 ml.) to make strongly alkaline. Pour the alkali down the side of the Kjeldahl flask, so that it does not mix with the acid at once.

(h) Connect the Kjeldahl to the other end of the condenser tube and mix the contents by shaking. Apply heat and distill until at least 150 ml. of distillate have been collected.

(i) Titrate the contents of the receiving flask with 0.25 N NaOH solution using 3 or 4 drops of indicator.

(j) Conduct a blank determination on the reagents, simultaneously with the sample and similar in all respects.

(k) Determine moisture in the ground sample as directed in A.O.C.S. Official Method Aa 3-38, Ba 2-38.

C. *Calculation*

$$\text{Ammonia, } NH_3\ (\%) = \frac{(B\text{-}S) \times N \times 0.017032 \times 100}{\text{Weight of sample}}$$

Where B = ml. of alkali back titration of blank.
S = ml. of alkali back titration of sample.

Calculate to the desired moisture basis as directed in A.O.C.S. Official Method Aa 4-38, Section C substituting % ammonia (nitrogen or protein) for F.

per cent ammonia (NH_3) $\times$ 5.14 = per cent protein,
per cent nitrogen (N) $\times$ 6.25 = per cent protein.

6. Free Fatty Acid Content in Cottonseed

A.O.C.S. Official Method Aa 6-38

A. Scope—The determination of the free fatty acid content of the oil in the seed is one of the most interesting and difficult problems. Deterioration of cottonseed oil usually takes the form of liberation of fatty acids, so that the percentage of free fatty acids in an oil indicates the relative deterioration of that oil. The U.S.A. National Cottonseed Producers Association Trading Rules insist that any crude cottonseed oil which exceeds 3.25 per cent in free fatty acid content should be automatically graded "Off" in flavour regardless of its actual flavour and odour.

B. *Procedures*—(a) If necessary for effective hulling, reduce moisture content by drying 200 g. of the mixed sample for about 30 minutes at 100° to 105°C. in an air oven and cool. Grind the seed through the Bauer Mill with the mill opened so that the seed is only broken. Remove the meat from the hulls by screening on a 4- to 6-mesh screen. Grind the meats in a Universal food cutter using the 12-tooth blade. Re-grind the first material through the chopper. Complete separation of meats from hulls and good grinding are essential. Immediately mix the ground sample on a clean, dry surface, divide the mass into 4 quarters with a spatula.

(b) Place the entire sample of 40 to 50 g. on the asbestos mat in the extraction apparatus. Add 50 ml. of petrol-ether and allow all of it to percolate through the sample into a flask or beaker. Repeat with two 25 ml. portions of solvent.

(c) Evaporate the solvent from the oil on a water bath under a gentle stream of clean, dry air until free from petrol-ether.

(d) Weigh 7.05 g. of extracted oil into oil-sample bottle or flask. Add 30 ml. of neutral alcohol and 1 ml. of 0.05% phenolphthalein indicator. Titrate with 0.25N NaOH shaking vigorously until a faint pink colour is obtained which will persist for at least one minute.

C. *Calculation*—Free fatty acids, calculated as oleic (%) = ml. of 0.25N NaOH solution used.

7. Residual Lint Content in Cottonseed

A.O.C.S. Official Method Aa 7-44

A. *Procedure*—(a) Use a well mixed sample free of foreign matter and broken seed particles.

(b) Weigh 50 g. of sample into moisture dish and dry in forced draft oven for 30 minutes at 130° ± 3°C. If the sample contains more than 14 per cent moisture, continue the drying for an additional 30-minutes period.

(c) Treat the dried sample as directed in A.O.C.S. Official Method Aa 4-38, Section A, Paragraphs b and c beginning with "Absorb into.....scorched", except that 2 ml. of concentrated HCl are used for normal seed and 1.0 ml. for delinted seed.

(d) Transfer the treated (fumed) seed to the sieve. Brush the seed carefully, using the round brush, with a rotating or circular motion until all the lint has been removed from the seed and has passed through the screen. It has been found that a 1-minute brushing period usually removes all the lint when the delinting machine is used.

(e) Transfer all the delinted seed to a tared moisture dish. With the cover removed, dry the seed in a forced draft oven at 101°±1°C. for 12 to 16 hrs. (overnight). Remove from the oven, cover immediately, cool to room temperature in a desiccator and weigh.

(f) Determine the moisture in the original seed as directed in A.O.C.S. Official Method Aa 3-38.

Note—The original moisture in the cottonseed and the weight of dry delinted seed operations (e) and (f) are determined in the same oven at the same time.

B. *Calculation*—The residual lint, that is, the lint remaining on the seed, is conventionally calculated to an 8 per cent moisture basis.

$$\text{Residual lint, 8\% moisture basis (when 50 g. sample is used), (\%)} = \frac{2(A\text{-}B) - C}{0.92}$$

Where A = weight of sample (50 g.) from A, b
B = do. dry delinted seed from A, e
C = moisture in original cottonseed from A, f.

8. Ash in Cottonseed Cake and Meal

A.O.C.S. Official Method Ba 5-49

A. Procedure—(a) Weigh 2 g. of well mixed sample ground to a uniform fineness of about 20 mesh into the previously heated and tared combustion capsule. Place in a muffie furnace previously heated to 600°C and maintain at this temperature (±15°C.) for 2 hr.

(b) Transfer capsule to a desiccator, cool to room temperature and weigh immediately thereafter.

B. *Calculation*—(Report to nearest 0.1%)

$$\text{Ash } (\%) = \frac{\text{Weight of ash} \times 100}{\text{Weight of sample}}$$

9. Crude Fibre in Cottonseed Cake and Meal

A.O.C.S. Official Method Ba 6-49

A. *Definition*—This method determines as crude fibre the loss on incineration of the dried residue remaining after digesting the sample with dilute H_2SO_4 and NaOH as specified for the test.

B. *Procedure*—(a) Extract 2 g. of the air dry material with ethyl or petroleum ether, or use residue from oil determination (A.O.C.S. Official Method Ba 3-38) and transfer residue, together with about 0.5 g. of asbestos, to digestion flask.

(b) Add 200 ml. of the boiling H_2SO_4 solution; immediately connect digestion flask with condenser and heat. (It is essential that contents of flask come to boiling within one minute and that the boiling continue briskly for exactly 30 minutes). Rotate flask about every 5 minues in order to mix charge thoroughly. Take care to keep material in contact with solution. (A blast of air conducted into flask will serve to reduce frothing of liquid).

(c) After 30 minutes remove flask, filter immediately through filtering cloth in fluted funnel and wash with boiling water until washings are no longer acid.

(d) Bring a quantity of NaOH solution to boiling and keep at this temperature under reflux condenser until used. Wash charge and asbestos back into flask with 200 ml. of the boiling NaOH solution using wash bottle marked to deliver 200 ml. The boiling NaOH solution is conveniently transferred to the wash bottle by means of bent tube through which liquid is forced by blowing into tube connected with top of reflux condenser attached to the NaOH flask.

(e) Connect flask with condenser and boil exactly for 30 minutes, so timing the boiling with the alkali that contents of different flasks

will reach boiling point about 3 minutes apart, which permits sufficient time for filtration.

(f) After 30 minutes remove flask and immediately filter through Gooch prepared with asbestos mat, through alundum crucible, or through the filtering cloth in fluted funnel. If the filtering cloth is used, thoroughly wash residue with boiling water and transfer it to Gooch crucible prepared with thin but close layer of ignited asbestos.

(g) After thorough washing with water, wash with about 15 ml. of alcohol.

(h) Dry crucible and contents in oven at 110°C. to constant weight. Cool in an efficient desiccator and weigh.

(i) Incinerate contents of cruicible in electric muffle furnace (500° to 550°C.) or over Meker burner at dull red heat until carbonaceous matter has been consumed (usually about 20 minutes). Cool to room temperature in desiccator and weigh.

C. *Calculation*

$$\text{Crude fibre } (\%) = \frac{\text{Loss in weight} \times 100}{\text{Weight of sample}}$$

10. Gossypol Content in Cottonseed and Meal

A.O.C.S. Official Method Ba 7-49

A. *Preparation of sample*—(a) Cottonseed. Grind the seed through the Bauer Mill with the mill opened, so that the seed is only broken. Separate the meats from the hulls and lint. Then grind the meats through a Wiley mill using the 1 mm. sieve.

(b) Cottonseed meals. Grind through the 1 mm. screen in a Wiley mill.

Caution—Do not preheat the cottonseed and avoid heating samples during grinding.

B. *Procedure*—(a) Weigh accurately sufficient samples to contain about 2.5 mg. of total free gossypol or gossypol-like substances into a glass-stoppered 250 ml. Erlenmeyer flask. This will be 0.25-0.30 g. of uncooked meats and unheated solvent-extracted meals, and 1.0-1.5 g. of hydraulic, screw-pressed and heated solvent-extracted meals.

(b) Cover the bottom of the flask with 6 mm. diam. solid glass beads and add 50 ml. of the aqueous acetone by pipette. Stopper the flask and shake on a mechanical shaker for one hour at room temperature at such a rate that the sample material which collects

around the top of the flask will be constantly washed back into the solution.

(c) Filter through a dry 11 cm. paper of medium retentivity into a small glass-stoppered flask, discarding the first portion of the filtrate. Place a watchglass over the funnel to reduce evaporation.

(d) Pipette duplicate 2 ml. aliquots of the filtrate into 25 ml. volumetric flasks. To one aliquot add 3 ml. of the acetic acid solution and make to volume with the 80 per cent isopropanol. This is the gossypol blank. To the other aliquot add 3 cc. of the *p*-anisidine solution and heat in a water-bath (with flask loosely stoppered) at 60°C. for half an hour. Cool to room temperature and make to volume with 80 per cent isopropanol.

(e) At the same time run a reagent blank containing 2 ml. of the aqueous acetone and 3 ml. of the *p*-anisidine solution parallel with the sample. After heating for half an hour in water-bath at 60°C., cool to room temperature and make to volume with 80 per cent isopropanol.

(f) Prepare a solvent blank consisting of 2 ml. of the aqueous acetone and 3 ml. of the acetic acid solution, made to 25 ml. volume with 80 per cent isopropanol.

(g) Determine the per cent transmission of the sample solution designed as T_1, setting the instrument at 100 per cent transmission with the reagent blank.

(h) Determine the per cent transmission of the gossypol blank, designated as T_2, using the solvent blank to set the instrument at 100 per cent transmission.

(i) Calculate the value $\frac{T_1}{T_2}$ and read the milligrams of gossypol in the solution from the calibration graph.

C. *Preparation of Calibration Graph*—(a) Pipette duplicate 2 ml. aliquots of the diluted standard solutions into a series of 25 ml. volumetric flasks. Develop and measure colour as prescribed for the sample B, (d) to (i) inclusive.

(b) Calculate the value $\frac{T_1}{T_2}$ for each concentration of gossypol. Plot on semi-logarithmic paper the value $\frac{T_1}{T_2}$ against the corresponding milligrams of gossypol in the 25 ml. volumetric flask. If semi-logarithmic paper is not available, regular co-ordinate paper may be used by plotting logarithmic $\frac{T_2}{T_1}$ against mg. of gossypol.

D. *Calculation*

$$\text{Gossypol (\%)} = \frac{\text{Mg. gossypol, [B (i)]} \times 2.5}{\text{Weight of sample [B (a)]}}$$

11. Sampling Cottonseed Oil and Hydrogenated Fat

A.O.C.S. Official Method C 1-47

A. *Size and number of sample*—(a) The general procedure is to draw a number of portions from the bulk quantity or a number of portions from all or several packages, composite these, mix thoroughly and distribute representative portions into suitably sized air-tight containers for the laboratory sample.

(b) A gross sample in the proportion of not less than 50 lb. per 60,000 lb. cargo is required of all tank cars and a minimum of 50 lb. for all other bulk oil quantities such as in ships or shore tanks.

(c) A gross sample in the proportion of not less than 20 lb. for each 100 barrels or equivalent quantity is required when drums, tierces, barrels, and other packages are sampled.

(d) In the case of edible fats and oils, the minimum size for each laboratory sample is 2 lb. for fats and 28 lb. for oils. If refining or bleaching tests are required the minimum quantity is about one gallon.

B. *Procedure*—(a) *Loaded Tanks or Tank Cars—Liquid Contents* (Official Method of the National Cottonseed Products Association)

(i) Lower the official trier vertically through the oil at a uniform rate with the bottom valve completely open so that 10 to 15 seconds will be required to reach the bottom of the car. (See Note below). Close the bottom valve and withdraw the tube.

Note—It is necessary that as the trier is lowered into the oil, the rate be slow enough so that the level of the oil inside and outside of the trier remain the same. Otherwise, an unduly large portion will be drawn from the bottom which is likely to contain a considerable concentration of moisture and settlings.

(ii) Take several portions in this manner and then proceed as directed under :

Collect the sample in a clean and dry container and protect from dirt, water, or other contamination. Mix the entire sample thoroughly so as to obtain uniform distribution of moisture, meal, and any other impurities and then fill 3 clean and dry 1 gallon containers.

(b) *Loaded Tanks or Tank Cars—Solid Contents*

(i) Solid material cannot be correctly sampled in tanks or tank cars. If possible, the material should be liquified and then sampled.

(ii) When necessary to sample solid material, use the designated trier and withdraw several portions from the car taken vertically and obliquely toward the ends of the car. The trier should pass through the stock until it touches the sides of the car so that a complete core will be taken. Soften (but do not melt) and mix all portions thoroughly before distributing into laboratory sample containers.

12. Refining Loss in Cottonseed Oil

A.O.C.S. Official Method Ca 9a-41

A. *Scope*—Crude cottonseed oil is sold primarily on the basis of refining loss and colour of the refined oil. The colour of the refined oil is roughly parallel to the free fatty acid content of the crude oil. Crude oil of "Prime" grade when refined by the official method must produce an oil with colour not in excess of 7.6 red units on the Lovibond scale.

B. *Preparation of sample*—The sample container must be vigorously shaken and the sample thoroughly mixed in order to incorporate and uniformly distribute meal or other sediments. If the oil is cold, heat to 20°C. before shaking. Inspect the inside of the can to be sure that no sediment remains clinging to the sides or bottom. If any sediment is found, remove it completely and incorporate thoroughly with the oil. The uniform incorporation and distribution of settlings and suspended matter are very significant, in determining the accuracy of the final refining loss.

C. *Determination of free fatty acids*

(a) *Normal coloured oils*—(i) Weigh 7.05 g. of well mixed sample into 115 ml. (4 oz.) oil sample bottle or 250 ml. Erlenmeyer flask and add 50 ml. of neutral alcohol containing 0.05 per cent phenolphthalein.

(ii) Warm in a water bath and titrate with 0.25 N NaOH solution until a permanent pink colour is obtained which persists after 30 seconds of vigorous shaking.

(iii) Free fatty acid (%) = ml. of 0.25N NaOH solution used.

(b) *Dark coloured oils*—(i) The procedure is identical with that described under (a) except that a solvent is used consisting of 99 per cent isopropanol, containing 0.025 per cent Dr. Grubler's Aniline Blue.

D. Procedure—(i) Refer to the Table 16 for specific details such as temperature, time intervals, etc.

Cauton—Samples must not be allowed to come in contact with copper since the colour may thereby be affected.

(ii) Weigh 500 g. of thoroughly mixed sample into tared refining cup and allow to settle to permit the air to escape. Remove any

persisting foam before adjusting the final weight. Place in the refining machine, fill the water bath to the specified height, and adjust the temperature of water and oil to the designated cold temperature (See Table 16).

(iii) Refer to section G and Table 17 to determine the correct amount of alkali to be added depending upon the type of oil and free fatty acid content. The designated amounts may be weighed into 50 ml. beakers and kept covered with watch glasses until ready to add to the sample. If beakers are used, compensation should be made for the lye which does not drain.

(iv) With the agitators running at high speed, add the alkali to each cup, draining as completely and quickly as possible. Continue high speed agitation and maintain the cold temperature within the required limits as directed in Table 16.

(v) Change the agitation to slow speed and increase the bath to the hot temperature. This temperature change should be completed in about one minute. Continue the agitation as directed in Table 16. At the end of the stirring period, the oil sample must itself be at the specified temperature. Adjust the temperature of the water bath to obtain the correct final oil temperature.

(vi) Discontinue agitation, remove paddles and return as much of the sample clinging to the paddles as possible. Allow the samples to set at the hot temperature for one hour. Cool the bath and allow to set for an additional 30 minutes. After this drain the bath and allow the samples to set for at least 12 hr., unless the oil has been maintained at the cold temperature (section E, Table 16). Cool again to this temperature for 30 minutes before pouring off.

(vii) Weigh the cup and contents and deduct this weight from the total weight at the start to obtain the evaporation loss.

(viii) Decant the refined oil into another tared refining cup and drain the soap stock for 30 minutes by inverting the soap stock cup into the refined oil cup. If the soap stock is too soft to drain, allow the cup to stand and pour off oil several times. Any floating foots decanted with the oil is recovered and returned to the main body of soap stock. The oil may be poured through a small strainer to collect the foots provided the screen is such that the oil is negligible.

(ix) Weigh the soap stock immediately to prevent any moisture loss. Melt the soap stock in a water bath at 75°±2°C. without stirring, for 30 minutes and then cool in cold water (see Table 16), for 15 minutes or until thoroughly chilled. Decant all possible oil into a tared beaker. Repeat remelting until not more than 1.5 g. is recovered and record the weight (to 0.1 g.) of all oil thus obtained. If the oil

is difficult to remove, due to soft soap stock or for any other reason, use a pipette.

(x) Weigh the refined oil and filter through the specified filter paper into a clean and dry container. Determine the colour as directed in A.O.C.S. Official Method Cc 13b-45. If a bleach test is required, it is determined on the filtered sample as directed in A.O.C.S. Official Method Cc 8a-49.

(xi) Cold pressed refined cottonseed oil is treated by adding 0.5 g. of filter-cel to the contents of the cup and agitating at 250±10 r.p.m. at room temperature for 2 to 3 minutes before filtering for the colour reading.

CAUTION : It is essential that the filtered oil obtained in (x) or (xi) above be absolutely clear, if not, refilter using whatman No. 12 filter paper.

TABLE 16—REFINING CONDITIONS OF COTTONSEED OIL

	Cottonseed oil			Cottonseed oil	
	Hydraulic	expeller		Hydraulic	expeller
Refining			*Clarification*		
Temperature of cold bath, °C.	20-24	20-24	Filter cell ..	0.5 g.	
R.P.M. in cold bath (±10)	250	250	Temperature ..	room	
Time in cold bath (min.)	15	45	r.p.m. (±10) ..	250	
Temperature of hot bath, °C.	63-67	63-67			
R.P.M. in hot bath (±5)	70	70	Remelting temp. of bath, °C.	73-77	73-77
Time in hot bath (min.)	12	12	Agitation	none	none
Final oil temperature, °C.	60-65	60-65			
Settling time in hot bath (min.)	60	60	Time in hot bath (min.)	30	30
Temperature of cooling bath, °C.	20-24	20-24	Temp. of cooling bath, °C.	20-24	20-24
Time in cooling bath (min.)	30	30	Time in cooling bath (min.)	15 or until firm	15 or until firm
Additional settling time (min. hr.)	12	12			
Chill soap stock (min.)	30	30	Drain (min.)	15	15
Drain soap stock (min.)	30	30			

F. *Calculation*—Calculate the refining loss by methods (i) and (ii) and report the average of these results which should agree within 0.25%.

$$\text{Refining loss (\%) (i)} = \frac{\text{Weight of crude oil—weight of refined oil}}{5}$$

$$\text{(ii)} = \frac{(A+B)-C}{5}$$

Where A = weight of soap stock,
B = evaporation loss, and
C = weight of alkali solution used.

G. *Lye requirements and Tables*—The maximum amount of the actual NaOH is calculated by the following formula :

$$\text{g. NaOH for 100 g. of hydraulic oil} = \frac{\%\ \text{F.F.A.}}{5.2} + 0.54 \quad \text{(i)}$$

$$\text{g. NaOH per 100 g. of expeller oil} = \frac{\%\ \text{F.F.A.}}{4.365} + 0.77 \quad \text{(ii)}$$

Example—Given sample of expeller cottonseed oil with a free fatty acid content (F.F.A.) of 2.2%.

Substituting in (ii) above,

$$\text{maximum actual NaOH} = \frac{2.2}{4.365} + 0.77 = 1.27 \text{ g.}$$

Three refinings are required (as per lye requirements in Table 17).

TABLE 17—LYE REQUIREMENTS—STRENGTH AND NUMBER OF REFININGS FOR COTTONSEED OIL *

	F.F.A. range	No. of refinings	Strength of alkali solutions °Be.
Hydraulic	0 - 1.5	2	80 % max. of 12°; 80 % max. of 14°
	1.6 - 3.0	3	80 % max. of 12°; 80 % max. of 16° max. of 16°
	3.1 - 4.0	2	max. of 14°; max. of 18°
	4.1 - 5.0	2	max. of 16°; max. of 20°
	5.1 - 7.5	2	max. of 18°; max. of 22°
	7.6 - 10.0	2	max. of 20°; max. of 24°
	10.1 - 15.0	2	max. of 20°; max. of 26°
	15.1 - 20.0	2	max. of 22°; max. of 28°
Expeller	0 - 3.0	3	80 % max. of 16°; 80 % max. of 20° max. of 20°
	3.1 - 5.0	2	max. of 16°; max. of 20°
	5.1 - 10.0	2	max. of 20°; max. of 26°
	Above 10.0	2	max. of 20°; max. of 30°

* Mehlenbachar, V. C. *Official and Tentative Methods* of the *American Oil Chemists' Society*, Second Edn., 1946.

a. $\frac{1.27\times80}{11.06}$ = 9.2% or 16° Be. of 46.0 g. per 500 g. of sample

b. $\frac{1.27\times80}{14.36}$ = 7.1% or 20°Be. of 35.5 g. per 500 g. of sample

c. $\frac{1.27\times100}{14.36}$ = 8.9% or 20°Be. of 44.5 g. per 500 g. of sample

13. Halphen Test for Identification of Cottonseed Oil

A.O.C.S. Official Method Cb 1-25

A. *Scope*—A very sensitive and reliable means of detecting cottonseed oil of 0.25 per cent or less is provided by the Halphen test. Of other oils Kapok oil responds to Halphen test as strongly as cottonseed but may be distinguished from cottonseed oil by means of Bessen test. After hydrogenation, cottonseed oil is no longer responsive to Halphen test.

B. *Procedure*—(i) Mix about 10 ml. of liquid sample in a 250×25 mm. test-tube with an equal volume of the reagent (a solution of one per cent sulphur in carbon disulphide with an equal volume of amyl alcohol). Shake and heat gently in hot water (70° to 80°C.) for a few minutes, with occasional shaking until the carbon disulphide is boiled off and the sample stops foaming.

Caution : Carbon disulphide vapours may be ignited with a hot bath or hot steam pipe.

(ii) Place the tube in the 110° to 115°C. bath and hold for 1 to 2 hr. A red colour at the end of this period indicates the presence of cottonseed oil.

(iii) If appreciable quantities of cottonseed oil are present, a positive reaction will be obtained in one hour or less in a water bath at or near the boiling temperature. For small amounts, a 2 hr. time interval may be necessary at a temperature of 110° to 115°C.

Note 1. The depth of colour is to a certain extent proportional to the amount of cottonseed oil present so that by comparing with samples containing known quantities of cottonseed oil, some idea of the amount can be obtained. However, at best this is only a rough approximation and subject to many doubts.

2. Hydrogenation and heating reduce the intensity of the reaction and may destroy it entirely.
3. Different lots of cottonseed oil may react giving different intensities.

14. Test for Refined and Bleached Oil

A.O.C.S. Official Method Cc 8a-49

A. *Definition*—This method determines the colour of the sample after treatment with a special bleaching earth under the conditions of this test.

B. *Procedure*—(i) Weigh 300 g. of sample into refining cup. Add official natural bleaching earth as specified on the label of the can.

(ii) Commence stirring at 250±10 r.p.m. and heat immediately to 120°C. for not more than 5 minutes. Do not allow the temperature of sample to drop below 105°C. during this period.

(iii) Filter through a dry filter paper. Allow a sufficient amount to pass through the filter paper to become entirely clear. Then collect a sufficient amount to fill a colour tube to the 133.35 mm. etched mark.

(iv) Determine the colour as directed in A.O.C.S. Official Method Cc 13b-45.

15. Wesson Method of Colour Determination

A.O.C.S. Official Method Cc 13b-45

A. *Scope*—This method determines the colour by comparison with glasses of known colour characteristics and applicable to all normal fats and oils provided no turbidity is present in the sample.

B. *Procedure*—(i) The sample must be absolutely clear, if not, filter through whatman No. 12.

(ii) Fill a colour tube with the sample to the 133.35 mm. etched mark. Adjust the temperature to 22±2°C. and read at this temperature. If the sample is not completely liquid at 22±2°C. heat to a temperature of not more than 10°C. above the point of complete melting.

NOTE: *Dark oils*: If the colour of the oil sample exceeds 40.0 red when using the regular 133.35 mm. column fill another tube to the 25.4 mm. mark and read the colour under the same conditions as described for the longer column. It is assumed that all colour determinations are read on a 133.35 mm. column. Only when the colour has been read on 25.4 mm. column, it is necessary to specify the length of column.

(iii) Place the tube containing the sample in the calorimeter and place along side of it such red and yellow glasses as are necessary to match the brightness of the oil, observing the colours of the glasses through the eyepiece.

C. *Refined and bleached cottonseed oil*—The ratio of yellow to red to be used in determining the colour is 10 yellow to 1 red up to 3.5 red ; 35 yellow for 3.5 red or above.

APPENDIX A

LIST OF APPARATUS AND REAGENTS

Apparatus

1. Cottonseed trier, corkscrew type that will take about 4 lb. of cottonseed at a single probe (A.O.C.S. Official Method Aa 1-38).
2. *Shaker-cleaner,* motor driven (Official Method Aa 1-38).
3. 1,000*g. capacity sample containers.*
4. *Scale* : (1) 10 lb. cap. sensitive to $\frac{1}{2}$ oz., (2) 100 lb. cap. sensitive to $\frac{1}{4}$ lb., (3) Balance, Torsion type 1,000 g. cap. sensitive to 0.1 g., (4) Balance weights 0.1 to 500 g.
5. *Forced draft oven* (A.O.C.S. Specification H 1-39).
6. *Aluminium moisture dishes* 2 in. $\times$ $\frac{3}{4}$ in., $3\frac{3}{4}$ in. $\times$ 1 in., 30-gauge with tight fitting slip-over covers.
7. *Desiccator* containing an efficient desiccant.
8. *Butt type extraction apparatus* (A.O.C.S. Official Method Aa 4-38).
9. *Whatman Filter papers* No. 2, 12 (110 mm., 330 mm.).
10. *Absorbent cotton,* free of petroleum-ether extract
11. *Airtight sample containers*
12. *Shallow, metal flat-bottom pans* for pre-drying (60 g. cap)
13. *Fuming oven* (Official Method Aa 4-38)
14. *Fuming vessels* (Official Method Aa 4-38)
15. *Electric muffle furnace or Meker type burner*
16. *Grinding mill and laboratory huller* (Bauer Bros. No. 148)
17. *Kjeldahl digestion and distillation apparatus*
18. *Kjeldahl flasks* 800 *ml.*
19. *Distillate receiving flasks* 500 ml.
20. *Extraction apparatus*
21. *Sieves* 4-6 *mesh and Tyler* 35 *mesh* (U.S. No. 40)
22. *Oil sample bottle*
23. *Universal Food Chopper No.* 1, with 12-tooth blade
24. *Round brush*
25. *Mechanical shaker*
26. *Photo-electric Calorimeter*
27. *Solid glass beads*
28. *Glass stoppered Erlenmeyer flasks* 250 *ml.*
29. *Pipettes,* volumetric 2, 3, 10 & 50 ml.
30. *Volumetric flasks* 25, 50 & 200 *ml.*
31. *Core sampler* for oils and liquid fats (A.O.C.S. Official Method C 1-47)
32. *Trier* for solid fats (A.O.C.S. Official Method C 1-47)
33. *Refining and bleaching apparatus* (A.O.C.S. Official Method Ca 9a-41)
34. *A.O.C.S. Standard Filter Cell*
35. *Test-tubes* 250 $\times$ 25 mm.
36. *Wesson type calorimeter* (A.O.C.S. Official Method Cc 13b-45)

The minimum standard set of Red and Yellow colour glasses consisting of : *Red* : 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1.0, 2.0, 2.5, 3.0, 3.5, 4.0, 5.0, 6.0, 7.0, 7.6, 8.0, 9.0, 10.0, 11.0, 12.0, 16.0, 20.0 ; *Yellow* : 1.0, 2.0, 3.0, 5.0, 10.0, 15.0, 20.0, 35.0, 50.0, 70.0

37. *Colour tubes of colourless glass* with a smooth flat polished bottom and of the following dimensions: Length 154 mm., I.D. 9 mm., O.D. 22 mm. The colour tubes are provided with two etched marks, one to indicate an oil column of 133.35 mm. and another to indicate an oil column of 25·4 mm.

Reagents

1. *Mercury or mercuric oxide* (A.C.S. grade)
2. *Sulphuric acid,* sp. gr., 1.84
3. *Zinc metal,* granular, 20 mesh
4. *Potassium or sodium sulphate* (A.C.S. grade)
5. *Potassium or sodium sulphide solution* 4 per cent in water
6. *Sodium hydroxide solution,* sp. gr., 1.5
7. *Sodium hydroxide* 0.25*N*, accurately standardised
8. *Sulphuric acid* 0.5*N* accurately standardised
9. *Methyl red indicator solution,* 0.1 per cent in ethyl alcohol or Alizarin Red solution, 0.3 per cent in distilled water
10. *Petroleum-ether* (A.O.C.S. Specification H2-41)
11. *Ethyl alcohol,* 95 per cent (U.S.S.D. Formulas 30 and 3A are permitted) or Isopropyl alcohol, 99 per cent. The alcohol must be neutralized with NaOH solution to a faint pink colour before adding the sample.
12. *Phenolphthalein indicator solution,* one per cent in 90 per cent alcohol
13. *Aqueous acetone*—Mix 700 ml. acetone (A.C.S. grade) and 300 ml. distilled water
14. *Isopropanol,* reagent grade, diluted to 80 per cent by volume with distilled water
15. *Glacial acetic acid* (A.C.S. reagent grade)
16. *Purified* p-*anisidine*. Dissolve 0.5 g. recrystallized *p*-anisidine in 80 per cent isopropanol. Add 1 ml. of glacial acetic acid and make to 50 ml. with 80 per cent isopropanol. Store in a brown bottle and prepare fresh daily.
17. *Acetic acid solution*: Dilute 1 ml. glacial acetic acid to 50 ml. with 80 per cent isopropanol
18. *Standard gossypol solutions*—(a) Standard stock solution: Weigh accurately 25 mg. of pure gossypol, transfer quantitatively to a 200 ml. volumetric flask with aqueous acetone, make to volume with aqueous acetone and mix thoroughly; (b) Diluted standard solution: Prepare 8 diluted standard solutions by pipetting the ml. of standard stock solution designated below into 50 ml. volumetric flasks. Dilute to volume with aqueous acetone and mix thoroughly.

Standard stock solution to be pipetted into 50 ml. volume flasks	Gossypol in 2 ml. of diluted solution
cc.	mg.
2	0.010
5	0.025
10	0.050
15	0.075
20	0.100
25	0.125
30	0.150
35	0.175
40	0.200

19. Prepare a solution of 1 per cent sulphur in carbon disulphide and then add equal volume of amyl alcohol.

APPENDIX B

CLASSIFIED LIST OF MANUFACTURERS AND SUPPLIERS OF EQUIPMENT FOR PROCESSING COTTONSEED

The following is a list of manufacturers and suppliers of equipment in foreign countries for processing cottonseed into oil, meal and by-product. In this list no guarantee of completeness is implied and no discrimination is intended. The Products of these firms are of general adoptability to the processing of many other oilseeds.

A. PREPARATION EQUIPMENT

1. Storage Systems

(a) *Seed houses, silos*

Butter Mfg. Co., 7384, East 13th Street, Kansas City, M.O.
Chicago Bridge & Iron Company, 2143, Mc.Cormack Bldg. Chicago, 4, Illinois
Cole, R. D. Mfg. Co., Newman, Georgia
Klein, J. B., Iron & Foundry Co., Oklahoma City, Oklahoma
Muskogee Iron Works, Muskogee, Oklahoma
Neff & Fry Co., Cambden, Ohio
Rust Engineering Co., Birmington, Ala

(b) *Seed cooling and ventilating systems*

American Metal Products Co., P. O. Box 7037, Sylvania Station, Ft. Worth, Texas
Buhler Bros. Uzwil, Switzerland
Fortworth Steel & Machinery Co., Box 1038, Fort Worth, Texas
M.I.A.G. (Muhlenbau und Industrie A. G.) Brusnwick, Germany
National Blow Pipe & Mfg., Co., Ltd., 738, Dryades St., New Orleans, La

2. Seed Cleaning Equipment

Atlanta Utility Works, East Point, Georgia
Bauer Bros. Co., The Springfield, Ohio
Boardman Co., The Oklahoma City, Oklahoma
Carter D. M. Mfg. Co., P. B. 108, Blakely, Ga
Carver Cotton Gin Co., East Bridgewater, Mass.
Davidson-Kennedy Co., Atlanta, Georgia
Huntley Mfg. Co., Silver Creek, New York
Link-Belt Co., 1116, Murphy Avenue, S. W., Atlanta, Georgia
National Blowpipe & Mfg. Co., Ltd., 738, Dryades St., New Orleans, La
Rose Downs & Thompson Ltd., Old Foundry, Hull, England
Sutton, Steele & Steele, Inc., Dallas, Texas

3. Delinters and Accessories

American Metal Products Co., P. B. 7037, Sylvania Station, Ft. Worth, Texas
Atlanta Utility Works, East Point, Georgia
Butters Mfg. Co., Atlanta, Georgia
Carver Cotton Gin Co., East Bridgewater, Mass.
Carver, Fred, S., 347, Hudson Street, New York 14
Continental Gin Co., Atlanta, Georgia
Fort Worth Steel & Machinery Co., P. B. 1038, Fort Worth, Texas
National Blowpipe & Mfg. Co., Ltd., 738, Dryades St., New Orleans, La
Rose Downs & Thompson Ltd., Old Foundry, Hull, England

4. Hulling Equipment

Atlanta Utility Works, East Point, Georgia
Bauer Bros., Co., The Springfield, Ohio
Carver Cotton Gin Co., East Bridgewater, Mass.
Chandler Construction Co., East Bridgewater, Mass.
Davidson-Kennedy Co., Atlanta, Georgia
Rose Downs & Thompson Ltd., Old Foundry, Hull, England

5. Separation Equipment

American Metal Products Co., P. B. 7037, Sylvania Station, Ft. Worth, Texas
Atlanta Utility Works, East Point, Georgia
Bauer Bros. Co., The Springfield, Ohio
Carver Cotton Gin Co., East Bridgewater, Mass.
Dawson Sheet Metal Works, Dawson, Georgia
Fort Worth Steel & Machinery Co., P. B. 1038, Fort Worth, Texas
Huntley Mfg., Silver Creek, New York
Rose Downs & Thompson Ltd., Old Foundry, Hull, England
Sutton, Steele & Steele, Inc., Dallas, Texas

6. Cracking, Flaking and Cooking Equipment

Allis-Chalmers Mfg. Co., Milwaukee 1, Wis.
Blaw-Knox Co., Blaw-Knox Division, P. B. 915, Pittsburgh, Pa.
Buckeye Iron & Brass Works, Dayton, Ohio
Buhler Bros., Uzwil, Switzerland
Davidson-Kennedy Co., Atlanta, Georgia
E. R. & F. Turner Ltd., Ipswich, England
French Oil Mill Machinery Co., Piqua, Ohio
Manlove Alliott & Co., Ltd., Nottingham, England
M. I. A. G. (Muhlenbau und Industrie, A. G.) Brunswick, Germany
Sprout, Waldron & Co., Sherman Street, Muncy, Pennsylvania
Wolf Co., The, 62, Commerce St., Chambersburg, Pa.

B. OIL EXTRACTION EQUIPMENT

1. Expeller Equipment

Anderson, V. D. Co., 1935, West, 96th St., Cleveland, Ohio
Fred Krupp, Essen, Germany
French Oil Mill Machinery Co., Piqua, Ohio
Fritz Muller. Esslingen, Germany
Rose Downs & Thompson Ltd., Old Foundry, Hull, England

2. Hydraulic Press

Briggs-Weaver Machinery Co., 309, North Market Street, Dallas 2, Texas
Buckeye Iron & Brass Works, Dayton, Ohio
Carver, Fred, S., 347, Hudson Street, New York 14
Davidson-Kennedy Co., Atlanta, Georgia
French Oil Mill Machinery Co., Piqua, Ohio
Manlove Alliott & Co., Ltd., Nottingham, England
Oriental Textile Mills, 610, Forsyth Building, Atlanta, Georgia
Wolf Co., 62, Commerce Street, Crambersburg Pa.

3. Solvent Extraction Equipment

Acme Coppersmithing & Machine Co., Oreland, Pa
Acme Industrial Equipment Corp., 9, Armory Street, Boston, Mass.
Allis-Chalmers Mfg. Co., Milwaukee 1 Wis.
American Metal Products Co., P. B. 7037, Sylvania Station, Ft. Worth. Texas

Anderson, V.D. Co., 1935, West 96th St., Cleveland, Ohio
Artisan Metal Products, Inc., 211, Congress Street, Boston, 10, Mass.
Badger, E. B. & Sons, 75 Pitts Street, Boston 14, Mass.
Bamag Ltd., Rickett Street, London, S. W. 6
Bartlett, C. O. & Snow Co., 6400, Harvard Avenue, Cleveland, 5, Ohio
Blaw-Knox Co., Blaw-Knox Division, P. B. 915, Pittsburgh, Pa.
Buffalo Foundry & Machine Co., 1575, Filmore Avenue, Buffalo
Bird Machine Co., South Walpole, Mass.
Carbide & Carbon Chemicals Corporation, 30E, 42nd St., New York 17
Christianson & Meyer, Harbourg Hambourg, Germany
Cole, R. D. Mfg. Co., Newman, Georgia
Detrix Corporation, 13005, Hillview Ave., Detroit, Michigan
Devine, J. P. Mfg., Co., Inc., Mt. Vernon, Illinois
Dupps, John J. Co., 408, American Building, Cincinnati, Ohio
French Oil Mill Machinery Co., Piqua, Ohio
Gorgia Scott & Son (London) Ltd., Levenbank, Leven-Fife, London
Hansa-Muhle A. G., Hamburg-Neuhof Alsterdam 3, Germany
Hicks, S. D. & Son Co., Inc., 51, East 42nd Street, New York 17
Koven, I. O. & Bros., Inc., 154, Ogden Avenue, Jersey City 7, N. J.
Leader Iron Works, Inc., 2500, North Jasper Street, Decatur, Illinois
Lee, Alan Porter Inc., 136, Liberty, St., New York
Lungi, Frankfurt-on-main, Western Germany
Lurmmus Co., 420, Lexington Avenue, New York 17
Newell Construction & Machinery Co., Cedar Rapids, Iowa
Philadelphia Coppersmithing Co., Poplar, Front, Brown Sts., Philadelphia, Pa.
Production Engineering Co., Inc., 420, Lexington Avenue, New York
Read Machinery Co., Inc., York, Pennsylvania
Rose Downs & Thompson Ltd., Old Foundry, Hull, England
Scott, Ernest & Co., Summer & Anawan, Fall River, Mass.
Sharples Corporation, the, 23rd and Westmoreland Sts., Philadelphia 3, Pa.
Sieck & Drucker, Inc., 9 South Clinton St., Chicago, Illinois
Stokes, F. J. Machine Co., 5010, Tabor Road, Olney Post Office, Philadelphia
Struthers-Wells Corporation, Warren, Pa.
Sulzer Bros. Ltd., Winterthur, Switzerland
Swenson Evaporator Co., 15649, Lathrop Avenue, Harvey, Ill.
Vulcan Copper & Supply Co., The, Cincinnatti, Ohio
Wolf Co., The, 62, Commerce St., Chambersburg, Pa.
Wurster & Sanger Inc., 5201, S. Kenwood Avenue, Chicago 15, Illinois

4. Miscellaneous Oil Mill Supplies (conveyors, lint & dust removal systems, meal grinders, cake breakers, etc.)

Allis-Chalmers Mfg. Co., Milwaukee 1, Wis.
American Metal Products Co., P. B. 7037, Sylvania Station, Ft. Worth, Texas
Bauer Bros. Co., The, Springfield, Ohio
Bird Machine Co., South Walpole, Mass.
Boardman Co., The, Oklahoma City, Oklahoma
Chandler Construction Co., East Bridgewater, Mass.
Continental Gin Co., Atlanta, Georgia
Duplex Mill & Mfg. Co., Springfield, Ohio
Fort Worth Steel & Machinery Co., P. B. 1038, Fort Worth, Texas
Fuller Co., Catasugua, Pennsylvania
Great Western Mfg. Co., Leavensworth, Kansas
Gruendler Crusher & Pulvarising Co., 2917, N., Market St.
Gum, B. F. Co., 441, South Chinton Street, Chicago, Illinois
Houston Belting & Supply Corp., 1115, Austin St., Houston, Texas
Industrial Machinery Co., P. B. 1259, 2300, South Main St., Ft. Worth, Texas
Jeffrey Mfg. Co., The, 924-99, North Fourth Street, Columbus, 16, Ohio
Lewis Supply Co., Memphis, Tenn.
Link-Belt Co., 1116, Murphy Avenue, S. W., Atlanta, Georgia
National Blowpipe & Mfg. Co., The, 738, Dryades St., New Orleans, La.
Patterson Foundry & Machine Co., The, East Liverpool, Ohio

Phelps, Hubert, Machinery Co., P. B. 1093, Little Rock, Arkansas
Riechman-Crosby Co., Front St., At Beale Ave. Memphis, Tenn.
Screw Conveyor Corporation, 711, Hoffmann St., Hammon, Indiana
Southern Engineering & Supply Co., P. B. 629, Vicksburg, Miss.
South Western Supply & Machine Works, Oklahoma City, Oklahoma
Sprout, Waldron & Co., Sherman Street, Muncy, Pennsylvania
Sutton, Steele & Steele, Inc., Dallas, Texas. 1, Texas
Well Machinery & Supply Co., Inc., 1629, Main St., Ft. Worth 1, Texas
Texas Belting & Supply So., Inc., 3313, Mc Kinney St., Houston, Texas

C. OIL PROCESSING EQUIPMENT

1. Refining, Bleaching and Deodorisation Equipment

Bamag Ltd., Rickett Street, London, S. W. 6
Blaw-Knox Co., Blaw-Knox Division, P. B. 915, Pittsburgh, Pa.
Delaval Separator Co., 165, Broadway, New York, 6, New York
Eclipse Fuel Engineering Co., 751, S. Main, Rockford, Illinois
Elliott Co., Jeannette, Pa.
Foster Wheeler Corporation, 165, Broadway, New York 1
Hicks, S. D. & Son Cl., Inc., 51, East, 42nd Street, New York
Kelogg, M. W. Co., Jersey City, New Jersey
Lee, Alan Porter, Inc., 136, Liberty St., New York
National Acme Co., The, 170, East, 131st Street, Cleveland, Ohio
Power Gas Corporation Ltd., The, Stockton-on-tees, England
Refining Inc., 30-30, Northern Boulevard, Long Island City, N. Y.
Rose Downs & Thompson Ltd., Old Foundry, Hull, England
Sharples Corporation, The, 23rd and Westmoreland, Stt., Philadephia 3
Sieck & Drucker, Inc., 9, South Clinton St., Chicago, Illinois
Struthers-Wells Corporation, Warren, Pa.
Wurster & Sanger, Inc., 5201, S. Kenwood Avenue, Chicago, 15, Illinois

2. Hydrogenation and Winterization Equipment and Accessories

Air Products, Incorporated, P. B. 1257, Chattangga, Tenn.
Blaw-Knox Co., Blaw-Knox Division, P. B. 915, Pittsburg, Pa.
Electric Heating Equipment Co., 32nd & Ludlow Streets, Philadelphia, 4, Pa.
Fost Wheeler Corporation, 165, Broadway, New York 1
Gas Industries Co., Pittsburgh, Pennsylvania
Girdler Corporation, The, Louisville 1, Kentucky
Harshaw Chemical Co., The, 1945, East 97th Street, Cleveland 6, Ohio
Kellog, M. W. Co., Jersey City, New Jersey
Krebs & Co., Zurich, Switzerland
Lee, Alan Porter, Inc., 136, Liberty St, New York
Power Gas Corporation Ltd., Stockton-on-tees, England
Schultz, W. F. H., Inc., 522, Fifth Avenue, New York
Seymour Mfg. Co., Ruffert Chemical Division, Seymour, Conn.
Sieck & Drucker, Inc., 9, South Clinton St., Chicago
Wurster & Sanger, Inc., 5201, S. Kenwood Avenue, Chicago, 15, Illinois

3. Shortening, Compound Cooking Fats Preparation Equipment

Blaw-Knox Co., Blaw-Knox Division, P. B. 915, Pittsburgh, Pa.
Eclipse Fuel Engineering Co., 751, S. Main Street, Rockford, Illinois
Elliott Co., Jeannette, Pa.
Foster Wheeler Corporation, 165, Broadway, New York
Girdler Corporation, The, Votator, Division, Louisville 1, Kentucky
Hicks, S. D. & Son Co., Inc., 51, East 42nd Street, New York 17
Lee, Alan Porter Inc., 136, Liberty St., New York
Sieck & Drucker, Inc., 9, South Clinton St., Chicago, Illinois
Struthers-Wells Corporation, Warren, Pa.

APPENDIX C

CLASSIFIED LIST OF COTTONSEED OIL MILLS IN THE INDIAN UNION*

1. *Alimchand Topandas Oil Mill,* Adoni (Andhra)
2. *Amalner Oil Mills,* Amalner (Bombay)
3. *Bhai Cursondas Oil Mill,* Amraoti (Madhya Pradesh)
4. *Bejaram Dedraj Oil Mill,* Dhulia, W. Khandesh Dist. (Bombay)
5. *Ghasiram Oil Mill,* Hyderabad
6. *Indian Cotton Oil Mill,* Navsari, Surat Dist. (Bombay)
7. *Kedia Ginning Factory & Oil Mill,* Khamgaon (Madhya Pradesh)
8. *Khamgaon Ginning Factory & Oil Mill,* Khamgaon (Madhya Pradesh)
9. *Mansingka Oil Mills,* Khandwa (Madhya Pradesh)
10. *Mansingka Oil Mills,* Amraoti (Madhya Pradesh)
11. *Rallis Oil Mills,* Guntakkal (Andhra)
12. *Sundatta Cottonseed Utilization Ltd.,* Hubli (Bombay)
13. *U. P. Oil Industries Ltd.,* Aishbagh, Lucknow (Uttar Pradesh)
14. *Venkatesh Cotton Oil Co.,* Khamgaon (Madhya Pradesh)
15. *Venkatesh Cotton Oil Co.,* Mancherial (Hyderabad)
16. *Venkatesh Cotton Oil Co.,* Parbahni (Hyderabad)

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